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Supporting Information

Synthesis of functionalized 2-pyridones via Michael addition and cyclization reaction of amines, alkynes and dialkyl acetylene dicarboxylates

Supporting Information

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EXPERIMENTAL SECTION

General Considerations. All reactions were carried out under an atmosphere of argon using standard Schlenk techniques unless otherwise noted. Column chromatography was carried out on silica gel (300–400 mesh) using a forced flow of eluent at 0.3–0.5 bar pressure. For TLC, silica gel GF254 was used and visualized by fluorescence quenching under UV light. Solvents were dried according to the standard procedure and were distilled prior to use.

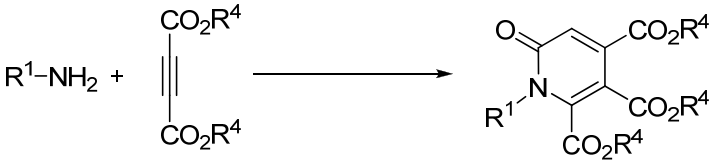
^{1}H NMR, ^{13}C NMR, ^{19}F NMR spectra were recorded at 400 MHz spectrometers. The chemical shifts for ^{1}H NMR were recorded in ppm downfield from tetramethylsilane (TMS) with the solvent resonance as the internal standard (2.05 ppm for CD_3COCD_3 or 7.26 ppm for $CDCl_3$). The chemical shifts for ^{13}C NMR were recorded in ppm downfield using the central peak of $CDCl_3$ (77.16 ppm) or CD_3COCD_3 (29.84 ppm) as the internal standard. Coupling constants (*J*) are reported in Hz and refer to apparent peak multiplications. The abbreviations *s*, *d*, *t*, *q*, and *m* stand for singlet, doublet, triplet, quartet, and multiplet in that order. All ^{13}C NMR spectra were proton decoupled.

Experimental Section.

R^1 = Alkyl; R^2 = H

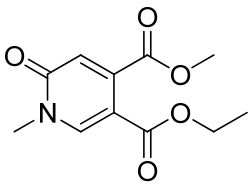
General Procedure for the Cyclization of Alkynes with Aliphatic Amines. Terminal alkyne (0.5 mmol for monamine, 1.0 mmol for diamine) and aliphatic

amines (0.5 mmol) were mixed in wet EtOH (2 ml) under argon atmosphere. The solution was stirred at room temperature for 24 h. Subsequently, dialkyl acetylene dicarboxylate (0.5 mmol for monamine, 1.0 mmol for diamine) was added and the solution was stirred at 78 °C under argon atmosphere. After completion of the reaction (as monitored by TLC), the solvent was removed under reduced pressure and the resulting residue was subjected to silica gel chromatography or recrystallized to give the desired product.



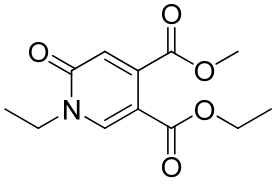
General Procedure for the Cyclization of Alkyne with Aromatic Amines.

A solution of aromatic amine (0.5 mmol), dialkyl acetylene dicarboxylate (1 mmol) and wet ethanol (2 mL) were stirred vigorously at room temperature for 10 min. Then the mixture was heated for 24 h. The progress of reaction was monitored by TLC (ether/ethyl acetate = 2:1). The solvent was removed under reduced pressure; the resulting residue was purified by silica gel chromatography. Further purification was done by recrystallization in mixture of ether/ethyl acetate (4:1).

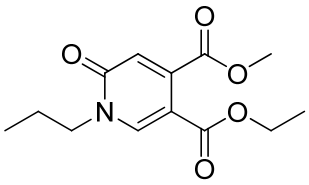


Analytical Data **3-Ethyl 4-methyl 1-methyl-6-oxo-1,6-dihydropyridine-3,4-dicarboxylate (4aa)**: white solid; 80.3 mg; 67% yield; mp 146 – 149 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10 (s, 1H), 6.47 (s, 1H), 4.22 (s, 2H), 3.84 (s, 3H), 3.53 (s, 3H), 1.26 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.7, 163.1, 161.9, 144.3, 144.1, 118.2, 107.3, 63.5, 52.9, 38.2, 14.2; HRMS (Q-TOF,

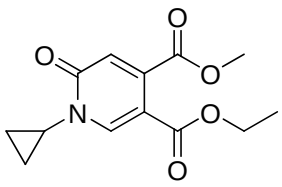
m/z) calcd for $C_{11}H_{13}NO_5Na$ $[M+Na]^+$ 262.0691, found 262.0701.



3-Ethyl 4-methyl 1-ethyl-6-oxo-1,6-dihydropyridine-3,4-dicarboxylate (4ab): white solid; 104 mg; 83% yield; mp 67 – 68 °C; ^{1}H NMR (400 MHz, CD_3COCD_3) δ 8.43 (s, 1H), 6.42 (s, 1H), 4.23 (s, 2H), 4.08 (s, 2H), 3.84 (s, 3H), 1.34 (s, 3H), 1.29 (s, 3H); ^{13}C NMR (100 MHz, CD_3COCD_3) δ 166.9, 163.5, 161.5, 145.3, 118.4, 107.4, 61.7, 52.9, 46.0, 14.6, 14.4; HRMS (Q-TOF, m/z) calcd for $C_{12}H_{15}NO_5Na$ $[M+Na]^+$ 276.0848, found 276.0853.

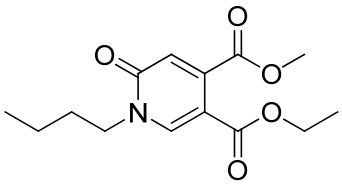


3-Ethyl 4-methyl 6-oxo-1-propyl-1,6-dihydropyridine-3,4-dicarboxylate (4ac): oil; 119 mg; 89% yield; ^{1}H NMR (400 MHz, CD_3COCD_3) δ 8.41 (s, 1H), 6.43 (s, 1H), 4.24 – 4.21 (m, 2H), 4.03 –4.01 (m, 2H) , 3.99 – 3.84 (m, 3H), 1.76 (d, J = 7.2 Hz, 2H), 1.28 (t, J = 6.9 Hz, 3H), 0.94 (t, J = 7.3 Hz, 3H); ^{13}C NMR (100 MHz, CD_3COCD_3) δ 167.1, 163.9, 161.6, 145.5, 144.8, 118.5, 107.1, 61.5, 52.9, 52.1, 22.9, 14.4, 11.1; HRMS (Q-TOF, m/z) calcd for $C_{13}H_{17}NO_5Na$ $[M+Na]^+$ 290.0997, found 290.1004.

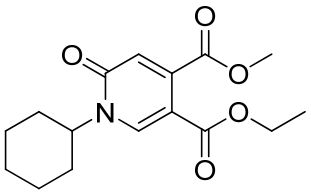


3-Ethyl 4-methyl 1-cyclopropyl-6-oxo-1,6-dihydropyridine-3,4-dicarboxylate (4ad): white solid; 104.6 mg; 79% yield; mp 115 – 116 °C; ^{1}H NMR (400 MHz, CD_3COCD_3) δ 8.20 (s,

1H), 6.39 (s, 1H), 4.24 (q, *J* = 6.5 Hz, 2H), 3.84 (s, 3H), 3.43 – 3.34 (m, 1H), 1.29 (t, *J* = 6.4 Hz, 3H), 1.11 (d, *J* = 6.0 Hz, 2H), 0.98 (s, 2H); ^{13}C NMR (100 MHz, CD_3COCD_3) δ 167.4, 164.1, 162.1, 144.8, 144.7, 118.3, 106.7, 61.7, 52.9, 33.6, 14.4, 6.9; HRMS (Q-TOF, m/z) calcd for $C_{13}H_{15}NO_5Na$ $[M+Na]^+$ 288.0855, found 288.0848.

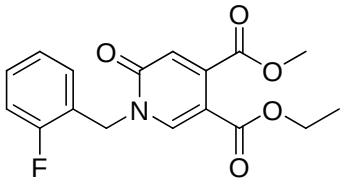


3-Ethyl 4-methyl 1-butyl-6-oxo-1,6-dihydropyridine-3,4-dicarboxylate (4ae): oil; 109.6 mg; 78% yield; ^{1}H NMR (400 MHz, CD_3COCD_3) δ 8.39 (s, 1H), 6.41 (s, 1H), 4.22 (m, 2H), 4.03 (s, 2H), 3.82 (s, 3H), 1.71 (s, 2H), 1.34 (s, 2H), 1.26 (s, 3H), 0.92 (s, 3H); ^{13}C NMR (100 MHz, CD_3COCD_3) δ 167.29, 163.9, 160.6, 145.5, 144.8, 118.4, 107.2, 61.7, 52.9, 50.4, 31.8, 20.3, 14.4, 13.9; HRMS (Q-TOF, m/z) calcd for $C_{14}H_{19}NO_5Na$ $[M+Na]^+$ 304.1161, found 304.1153.

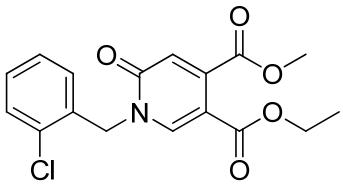


3-Ethyl 4-methyl 1-cyclohexyl-6-oxo-1,6-dihydropyridine-3,4-dicarboxylate (4af): white solid; 106.3 mg; 69% yield; mp 77 – 78 °C; ^{1}H NMR (400 MHz, CD_3COCD_3) δ 8.31 (s, 1H), 6.43 (s, 1H), 4.67-4.70 (t, *J* = 11.7 Hz, 1H), 4.24 (d, *J* = 3.3 Hz, 2H), 3.84 (s, 3H), 1.92 (d, *J* = 10.1 Hz, 4H), 1.70 (t, *J* = 11.3 Hz, 3H), 1.49 (dd, *J* = 24.3, 12.4 Hz, 2H), 1.28 (s, 4H); ^{13}C NMR (100 MHz, CD_3COCD_3) δ 167.4, 164.4, 161.1, 143.9), 141.5, 118.4, 107.3, 61.7, 56.2, 52.9, 32.4, 26.4, 25.7, 14.4; HRMS (Q-TOF, m/z) calcd for

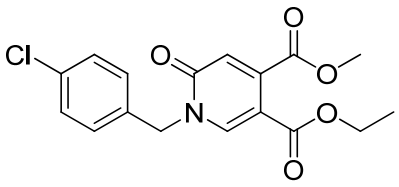
$C_{16}H_{21}NO_5Na$ $[M+Na]^+$ 330.1317, found 330.1310.



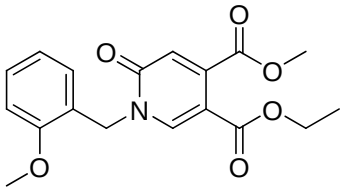
3-Ethyl 4-methyl 1-(2-fluorobenzyl)-6-oxo-1,6-dihydropyridine-3,4-dicarboxylate (4ag)*:* white solid; 114.7 mg; 69% yield; mp 106 – 117 °C; ^{1}H NMR (400 MHz, CD_3COCD_3) δ 8.52 (s, 1H), 7.35 (t, *J* = 6.4 Hz, 2H), 7.16 (dd, *J* = 15.4, 8.1 Hz, 2H), 6.48 (s, 1H), 5.31 (s, 2H), 4.26 (q, *J* = 7.1 Hz, 2H), 3.84 (s, 3H), 1.28 – 1.25 (m, 3H); ^{13}C NMR (100 MHz, CD_3COCD_3) δ 167.1, 163.8, 163.0, 161.5, 160.2, 145.7, 145.1, 131.1, 131.0, 125.3, 118.7, 116.3, 107.7, 61.8, 52.9, 47.94, 47.90, 14.4; 19F NMR (377 MHz, CD_3COCD_3) δ -119.4; HRMS (Q-TOF, m/z) calcd for $C_{17}H_{16}FNO_5Na$ $[M+Na]^+$ 356.0910, found 356.0897.



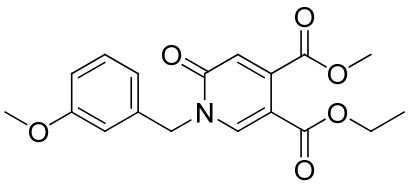
3-Ethyl 4-methyl 1-(2-chlorobenzyl)-6-oxo-1,6-dihydropyridine-3,4-dicarboxylate (4ah): white solid; 141.4 mg; 81% yield; mp 114 – 115 °C; ^{1}H NMR (400 MHz, CD_3COCD_3) δ 8.47 (s, 1H), 7.45 (s, 1H), 7.31 (s, 2H), 7.20 (s, 1H), 6.50 (s, 1H), 5.32 (s, 2H), 4.19-4.21 (d, *J* = 6.8 Hz, 2H), 3.84 (s, 3H), 1.23 (s, 3H); ^{13}C NMR (100 MHz, CD_3COCD_3) δ 166.9, 163.6, 161.4, 145.7, 145.6, 145.2, 134.2, 133.8, 130.49, 130.44, 0, 118.8, 107.6, 61.8, 53.0, 51.2, 14.4; HRMS (Q-TOF, m/z) calcd for $C_{17}H_{16}ClNO_5Na$ $[M+Na]^+$ 372.0615, found 372.0631.



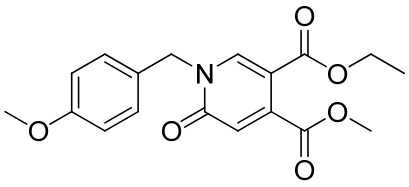
3-Ethyl 4-methyl 1-(4-chlorobenzyl)-6-oxo-1,6-dihydropyridine-3,4-dicarboxylate (4ai): white solid; 128.9 mg; 74% yield; mp 132 – 133 °C; ^{1}H NMR (400 MHz, CD_3COCD_3) δ 8.55 (s, 1H), 7.41 (dd, *J* = 19.1, 7.3 Hz, 4H), 6.50 (s, 1H), 5.27 (s, 2H), 4.23 (d, *J* = 6.0 Hz, 2H), 3.85 (s, 3H), 1.26 (t, *J* = 6.4 Hz, 3H); ^{13}C NMR (100 MHz, CD_3COCD_3) δ 166.6, 163.6, 161.5, 145.4, 145.1, 136.2, 134.2, 130.7, 129.5, 118.9, 107.9, 61.8, 53.0, 52.5, 14.4; HRMS (Q-TOF, m/z) calcd for $C_{17}H_{16}ClNO_5Na$ $[M+Na]^+$ 372.0615, found 372.0631.



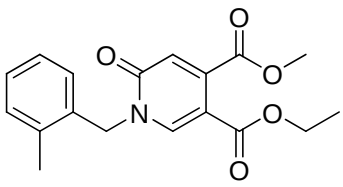
3-Ethyl 4-methyl 1-(2-methoxybenzyl)-6-oxo-1,6-dihydropyridine-3,4-dicarboxylate (4aj): white solid; 135.5 mg; 80% yield; mp 119 – 120 °C; ^{1}H NMR (400 MHz, CD_3COCD_3) δ 8.51 (s, 1H), 7.31 (s, 2H), 7.03 (d, *J* = 6.3 Hz, 1H), 6.93 (s, 1H), 6.43 (s, 1H), 5.16 (s, 2H), 4.23 (d, *J* = 2.4 Hz, 2H), 3.89 (s, 3H), 3.83 (s, 3H), 1.28 (s, 3H); ^{13}C NMR (100 MHz, CD_3COCD_3) δδ 167.2, 164.0, 161.6, 158.2, 146.2, 144.5, 131.7, 130.7, 121.3, 118.4, 111.6, 107.2, 61.7, 49,3, 14.4; HRMS (Q-TOF, m/z) calcd for $C_{18}H_{19}NO_6Na$ $[M+Na]^+$ 368.1110, found 368.1100.



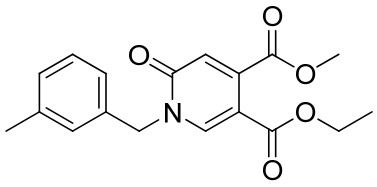
3-Ethyl 4-methyl 1-(3-methoxybenzyl)-6-oxo-1,6-dihydropyridine-3,4-dicarboxylate (4ak): white solid; 124.2 mg; 72% yield; mp 66 – 67 °C; ^{1}H NMR (400 MHz, CD_3COCD_3) δ 8.44 (s, 1H), 7.21 (d, J = 6.2 Hz, 1H), 6.93 (d, J = 9.9 Hz, 1H), 6.89 (s, 1H), 6.82 (s, 1H), 6.47 (s, 1H), 5.19 (s, 2H), 4.15 (s, 2H), 3.80 (s, 3H), 3.72 (s, 3H), 1.19 (s, 3H); ^{13}C NMR (100 MHz, CD_3COCD_3) δ 167.2, 163.8, 161.6, 160.9, 145.3, 144.9, 138.7, 130.7, 120.8, 118.8, 111.4, 107.8, 61.8, 55.5, 52.9, 52.8, 14.4; HRMS (Q-TOF, m/z) calcd for $C_{18}H_{19}NO_6Na$ $[M+Na]^+$ 368.1110, found 368.1100



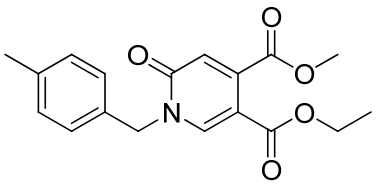
3-Ethyl 4-methyl 1-(4-methoxybenzyl)-6-oxo-1,6-dihydropyridine-3,4-dicarboxylate (4al): white solid; 104.3 mg; 61% yield; mp 97 – 98 °C; ^{1}H NMR (400 MHz, CD_3COCD_3) δ 8.43 (s, 1H), 7.31-7.34 (d, *J* = 8.7 Hz, 2H), 6.84-6.91 (d, *J* = 8.7 Hz, 2H), 6.44 (s, 1H), 5.13 (s, 2H), 4.14-4.19 (q, *J* = 7.1 Hz, 2H), 3.79 (s, 3H), 3.72 (s, 3H), 1.18-1.25 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, CD_3COCD_3) δ 167.2, 163.9, 161.7, 160.5, 145.1, 144.9, 130.6, 129.1, 118.8, 114.9, 107.7, 61.8, 55.5, 53.0, 52.5, 14.4; HRMS (Q-TOF, m/z) calcd for $C_{18}H_{19}NO_6Na$ $[M+Na]^+$ 368.1110, found 368.1098.



3-Ethyl 4-methyl 1-(2-methylbenzyl)-6-oxo-1,6-dihydropyridine-3,4-dicarboxylate (4am): white solid; 120.7 mg; 74% yield; mp 122 – 123 °C; ^{1}H NMR (400 MHz, CD_3COCD_3) δ 8.31 (d, *J* = 5.2 Hz, 1H), 7.20-7.24 (d, *J* = 13.0 Hz, 3H), 7.04 (d, *J* = 5.3 Hz, 1H), 6.50-6.52 (d, *J* = 5.3 Hz, 1H), 5.26 (s, 2H), 4.18 (d, *J* = 12.4 Hz, 2H), 3.84-3.87 (d, *J* = 5.3 Hz, 3H), 2.35 (d, *J* = 5.3 Hz, 3H), 1.22 – 1.25 (m, 3H); ^{13}C NMR (100 MHz, CD_3COCD_3) δ 166.1, 163.9, 161.8, 145.2, 131.4, 128.8, 128.3, 127.1, 118.6, 107.3, 61.8, 53.0, 50.6, 19.1, 14.3; HRMS (Q-TOF, m/z) calcd for $C_{18}H_{19}NO_5Na$ $[M+Na]^+$ 352.1161, found 352.1176.

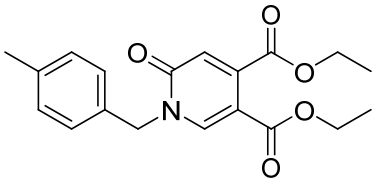


3-Ethyl 4-methyl 1-(3-methylbenzyl)-6-oxo-1,6-dihydropyridine-3,4-dicarboxylate (4an): white solid; 129.2 mg; 79% yield; mp 118 – 119 °C; ^{1}H NMR (400 MHz, CD_3COCD_3) δ 8.48 (s, 1H), 7.28 – 7.04 (m, 4H), 6.43 (s, 1H), 5.22 (s, 2H), 4.20-4.23 (s, 2H), 3.84 (s, 3H), 2.29 (s, 3H), 1.26 (s, 3H); ^{13}C NMR (100 MHz, CD_3COCD_3) δ 167.1, 164.6, 162.2, 145.4, 137.2, 129.5, 125.9, 118.8, 107.8, 61.8, 52.98, 52.92, 21.3, 14.4; HRMS (Q-TOF, m/z) calcd for $C_{18}H_{19}NO_5Na$ $[M+Na]^+$ 352.1161, found 352.1176.

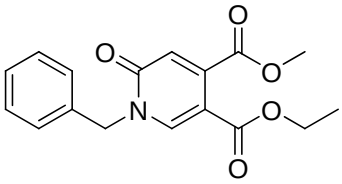


3-Ethyl 4-methyl 1-(4-methylbenzyl)-6-oxo-1,6-dihydropyridine-3,4-dicarboxylate (4ao): white solid; 127.6 mg; 78% yield; mp 128 – 129 °C; ^{1}H NMR (400 MHz, CD_3COCD_3) δ 8.47 (s, 1H), 7.28-7.30 (d, *J* = 8.0 Hz, 2H), 7.16-7.18 (d, *J* = 7.9 Hz, 2H), 6.49 (s, 1H), 5.22 (s, 2H), 4.19-4.21 (q, *J* = 7.1 Hz, 2H), 3.84 (s, 3H), 2.29 (s, 3H), 1.24-1.27 (t, *J* =

7.1 Hz, 3H); ^{13}C NMR (100 MHz, CD_3COCD_3) δ 166.7, 163.7, 161.3, 145.3, 144.9, 138.5, 134.3, 130.2, 129.0, 118.8, 107.5, 61.7, 52.9, 52.7, 21.0, 14.4; HRMS (Q-TOF, m/z) calcd for $C_{18}H_{19}NO_5Na$ $[M+Na]^+$ 352.1161, found 352.1167.

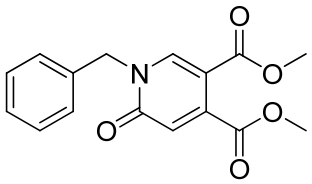


Diethyl 1-(4-methylbenzyl)-6-oxo-1,6-dihydropyridine-3,4-dicarboxylate (4ap): white solid; 116.5 mg; 68% yield; mp 121 – 122 °C; ^{1}H NMR (400 MHz, CD_3COCD_3) δ 8.46 (s, 1H), 7.28-7.30 (d, *J* = 8.0 Hz, 2H), 7.17-7.19 (d, *J* = 7.9 Hz, 2H), 6.48 (s, 1H), 5.22 (s, 2H), 4.31 (q, *J* = 7.1 Hz, 2H), 4.22 (q, *J* = 7.1 Hz, 2H), 2.30 (s, 3H), 1.31 (t, *J* = 7.1 Hz, 3H), 1.26 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, CD_3COCD_3) δ 166.1, 163.9, 161.7, 145.27, 145.23, 138.5, 134.3, 130.2, 128.9, 118.8, 107.8, 62.4, 61.7, 52.6, 21.0, 14.4, 14.2; HRMS (Q-TOF, m/z) calcd for $C_{19}H_{21}NO_5Na$ $[M+Na]^+$ 366.1317, found 366.1311.

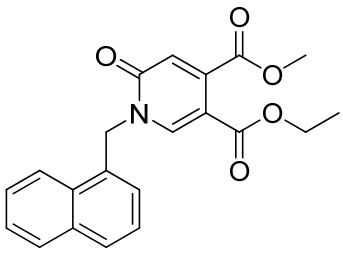


3-Ethyl 4-methyl 1-benzyl-6-oxo-1,6-dihydropyridine-3,4-dicarboxylate (4aq): white solid; 146.7 mg; 93% yield; mp 130 – 131 °C; ^{1}H NMR (400 MHz, DMSO) δ 8.67 (s, 1H), 7.28-7..35 (dd, *J* = 17.2, 6.8 Hz, 5H), 6.58 (s, 1H), 5.23 (s, 2H), 4.18-4.24 (q, *J* = 7.1 Hz, 2H), 3.80 (s, 3H), 1.18-1.26 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, DMSO) δ 167.5, 164.2, 161.9, 146.5, 145.0, 137.6, 130.0, 129.1, 129.0,

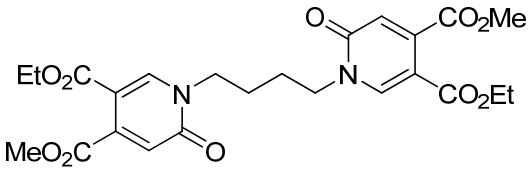
118.9, 107.5, 62.4, 54.0, 53.2, 15.3; HRMS (Q-TOF, m/z) calcd for $C_{17}H_{17}NO_5Na$ $[M+Na]^+$ 338.1004, found 338.1020.



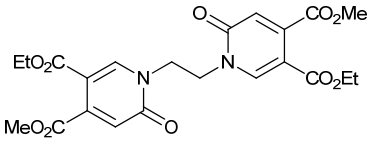
Dimethyl 1-benzyl-6-oxo-1,6-dihydropyridine-3,4-dicarboxylate (4ar): white solid; 115.9 mg; 77% yield; mp 125 – 127 °C; ^{1}H NMR (400 MHz, CD_3COCD_3) δ 8.49 (s, 1H), 7.42 – 7.29 (m, 5H), 6.50 (s, 1H), 5.27 (s, 2H), 3.84 (s, 3H), 3.75 (s, 3H); ^{13}C NMR (100 MHz, CD_3COCD_3) δ 167.1, 163.8, 161.7, 145.4, 145.0, 137.3, 129.6, 128.9, 128.8, 118.9, 107.4, 53.03, 53.00, 52.4.; HRMS (Q-TOF, m/z) calcd for $C_{16}H_{15}NO_5Na$ $[M+Na]^+$ 324.0848, found 324.0843.



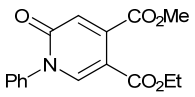
3-Ethyl 4-methyl 1-(naphthalen-1-ylmethyl)-6-oxo-1,6-dihydropyridine-3,4-dicarboxylate (4as): white solid; 174 mg; 95% yield; mp 117 – 119 °C; ^{1}H NMR (400 MHz, CD_3COCD_3) δ 8.34 (s, 1H), 8.11 (d, J = 7.9 Hz, 1H), 7.94-7.98 (dd, J = 15.7, 8.0 Hz, 2H), 7.61 – 7.51 (m, 3H), 7.39-7.40 (d, J = 6.9 Hz, 1H), 6.58 (s, 1H), 5.76 (s, 2H), 4.12-4.18 (q, J = 7.0 Hz, 2H), 3.85 (s, 3H), 1.17-1.20 (t, J = 7.0 Hz, 3H).; ^{13}C NMR (100 MHz, CD_3COCD_3) δ 167.1, 163.7, 161.7, 144.9, 144.6, 129.81, 129.80, 129.73, 129.70, 127.13, 127.1, 123.9, 118.7, 107.7, 61.7, 53.0, 49.8, 14.3; HRMS (Q-TOF, m/z) calcd for $C_{21}H_{19}NO_5Na$ $[M+Na]^+$ 388.1161, found 388.1158.



3-Diethyl 4-dimethyl 1,1'-(butane-1,4-diyl)bis(6-oxo-1,6-dihydropyridine-3,4-dicarboxylate) (4at): white solid; 0.305 mg; 61% yield; mp 216 – 217 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.11 (s, 2H), 6.51 (s, 2H), 4.26-4.28 (d, *J* = 6.6 Hz, 4H), 4.01 (s, 4H), 3.88 (s, 6H), 1.79 (s, 4H), 1.32 (d, *J* = 26.7 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.5, 163.0, 161.5, 144.1, 143.5, 118.7, 107.8, 61.6, 53.0, 49.8, 26.1, 14.2; HRMS (Q-TOF, m/z) calcd for $C_{24}H_{28}N_2O_{10}Na$ $[M+Na]^+$ 527.1642, found 527.1649.

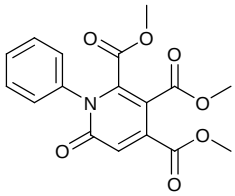


3-Diethyl 4-dimethyl 1,1'-(ethane-1,2-diyl)bis(6-oxo-1,6-dihydropyridine-3,4-dicarboxylate) (4au): white solid; 141 mg; 30% yield; mp 214 – 215 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (s, 2H), 6.56 (s, 2H), 4.32 (s, 4H), 4.22-4.27 (q, *J* = 7.1 Hz, 4H), 3.88 (s, 6H), 1.27-1.31 (t, *J* = 7.1 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.4, 162.4, 161.5, 144.9, 143.5, 118.7, 108.4, 61.8, 53.1, 48.8, 14.2; HRMS (Q-TOF, m/z) calcd for $C_{22}H_{24}N2O10Na$ $[M+Na]^+$ 499.1329, found 499.1335.

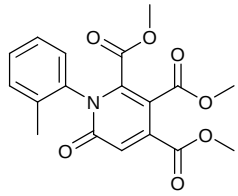


3-Ethyl 4-methyl 6-oxo-1-phenyl-1,6-dihydropyridine-3,4-dicarboxylate (4av): white solid; 176 mg; 28% yield (2 mmol); mp 123 – 124 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.17 (s, 1H), 7.50 – 7.52 (m, 3H), 7.37 (s, 2H), 6.63 (s, 1H), 4.28 (d, *J* = 4.1 Hz, 2H), 3.93 (s, 3H),

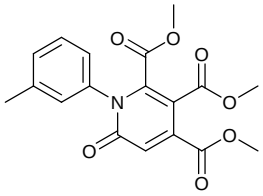
1.30 (t, J = 12.9 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.6, 162.6, 161.3, 144.0,139.4, 129.7, 126.3, 119.6, 107.6, 61.6, 53.1, 14.2; HRMS (Q-TOF, m/z) calcd for $C_{16}H_{15}NO_5Na$ $[M+Na]^+$ 324.0848, found 324.0846.



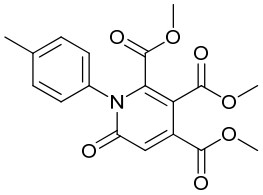
Trimethyl 6-oxo-1-phenyl-1,6-dihydropyridine-2,3,4-tricarboxylate (5aa): white solid; 138mg; 83% yield; mp 112 – 113 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48 (d, J = 8.0 Hz, 3H), 7.25 (d, J = 8.1 Hz, 2H), 6.84 (s, 1H), 3.91 (s, 3H), 3.80 (s, 3H), 3.48 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.6, 163.5, 161.2, 160.5, 145.6, 143.1, 136.2, 129.9, 129.3, 128.1, 121.3, 106.6, 53.1, 53.0, 52.9; HRMS (Q-TOF, m/z) calcd for $C_{17}H_{15}NO_7Na$ $[M+Na]^+$ 368.0746, found 368.0740.



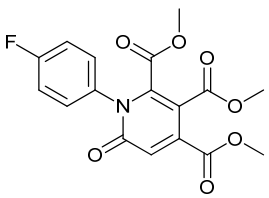
Trimethyl 6-oxo-1-o-tolyl-1,6-dihydropyridine-2,3,4-tricarboxylate (5ab): white solid; 131.1 mg; 73% yield; mp 113 – 114 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35-7.37 (dt, J = 8.0 Hz, 3H), 7.27 (d, J = 7.2 Hz, 1H), 6.83 (s, 1H), 3.92 (s, 3H), 3.79 (s, 3H), 3.46 (s, 3H), 2.16 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.7, 163.4, 161.1, 159.8, 145.8, 143.4, 136.5, 135.6, 135.4, 131.0, 130.2, 127.9, 126.7, 121.0, 106.3, 53.1, 52.9, 52.8, 17.4; HRMS (Q-TOF, m/z) calcd for $C_{18}H_{17}NO_7Na$ $[M+Na]^+$ 382.0903, found 382.0908.



Trimethyl 6-oxo-1-m-tolyl-1,6-dihydropyridine-2,3,4-tricarboxylate (5ac): white solid; 137.7 mg; 74% yield; mp 134 – 135 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 (t, *J* = 7.7 Hz, 1H), 7326-7.28 (d, *J* = 7.7 Hz, 1H), 7.04 – 7.05 (m, 2H), 6.83 (s, 1H), 3.91 (s, 3H), 3.80 (s, 3H), 3.50 (s, 3H), 2.39 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.7, 163.6, 161.3, 160.6, 145.6, 143.1, 139.5, 136.1, 129.1, 128.5, 124.9, 121.3, 106.5, 53.1, 53.0, 52.9, 21.2; HRMS (Q-TOF, m/z) calcd for $C_{18}H_{17}NO_7Na$ $[M+Na]^+$ 382.0903, found 382.0912.

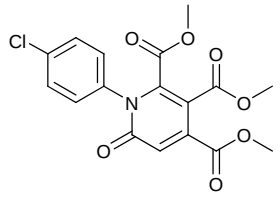


Trimethyl 6-oxo-1-p-tolyl-1,6-dihydropyridine-2,3,4-tricarboxylate (5ad): white solid; 168.8 mg; 90% yield; mp 124 – 125 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27-7.29 (d, *J* = 8.0 Hz, 2H), 7.11-7.13 (d, *J* = 8.1 Hz, 2H), 6.83 (s, 1H), 3.92 (s, 3H), 3.80 (s, 3H), 3.52 (s, 3H), 2.40 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.7, 163.6, 161.4, 160.9, 145.6, 143.2, 140.2, 133.6, 130.1, 127.8, 121.3, 106.6, 53.2 53.1, 53.0, 21.3; HRMS (Q-TOF, m/z) calcd for $C_{18}H_{17}NO_7Na$ $[M+Na]^+$ 382.0903, found 382.0906.

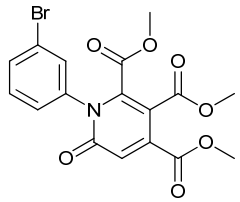


Trimethyl 1-(4-fluorophenyl)-6-oxo-1,6-dihydropyridine-2,3,4-tricarboxylate (5ae): white

solid; 134.7 mg; 71% yield; mp 160 – 162 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.23-7.27 (d, *J* = 8.0 Hz, 2H), 7.17-7.19 (d, *J* = 8.1 Hz, 2H), 6.81 (s, 1H), 3.92 (s, 3H), 3.81 (s, 3H), 3.55 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.6, 164.3, 163.5, 161.8, 161.3, 160.6, 145.6, 143.3, 132.1, 130.3, 130.2, 121.4, 116.7, 116.4, 106.8, 53.28, 53.026, 53.0; ^{19}F NMR (377 MHz, $CDCl_3$) δ -110.6; HRMS (Q-TOF, m/z) calcd for $C_{17}H_{14}FNO_7Na$ $[M+Na]^+$ 386.0652, found 386.0644.

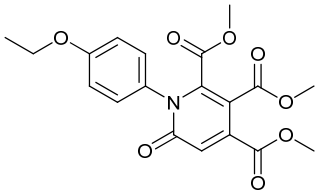


Trimethyl 1-(4-chlorophenyl)-6-oxo-1,6-dihydropyridine-2,3,4-tricarboxylate (5af): white solid; 139.8 mg; 71% yield; mp 150 – 151 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45-7.47 (d, *J* = 8.0 Hz, 2H), 7.21 (d, *J* = 8.1 Hz, 2H), 6.83 (s, 1H), 3.92 (s, 3H), 3.81 (s, 3H), 3.56 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.5, 163.5, 161.2, 160.4, 145.3, 143.3, 136.1, 134.7, 129.7, 129.6, 121.5, 106.8, 53.3, 53.2, 53.0; HRMS (Q-TOF, m/z) calcd for $C_{17}H_{14}ClNO_7Na$ $[M+Na]^+$ 402.0356, found 402.0347.

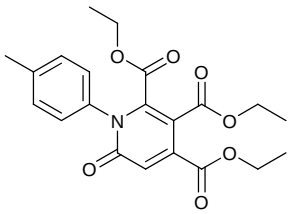


Trimethyl 1-(3-bromophenyl)-6-oxo-1,6-dihydropyridine-2,3,4-tricarboxylate (5ag): white solid; 145.5 mg; 69% yield; mp 151 – 152 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61-7.63 (d, *J* = 8.1 Hz, 1H), 7.39 (s, 1H), 7.45 (t, *J* = 8.0 Hz, 1H), 7.37 (d, *J* = 8.0 Hz, 1H), 6.84 (s, 1H), 3.92 (s, 3H), 3.81 (s, 3H), 3.56 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.5, 163.4, 162.3, 161.1, 160.3, 145.0, 143.2, 137.3, 131.3, 131.1, 130.4,

127.1, 122.5, 121.6, 107.1, 53.3, 53.2, 53.0; HRMS (Q-TOF, m/z) calcd for $C_{17}H_{14Br}NO_7Na$ $[M+Na]^+$ 445.9851, found 445.9858.

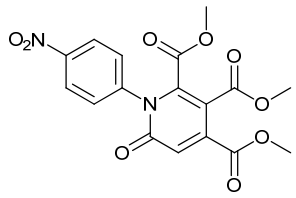


Trimethyl 1-(4-ethoxyphenyl)-6-oxo-1,6-dihydropyridine-2,3,4-tricarboxylate (5ah): white solid; 180.7 mg; 89% yield; mp 103 – 104 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.13-7.16 (d, *J* = 8.7 Hz, 2H), 6.94-6.96 (d, *J* = 8.7 Hz, 2H), 6.81 (d, *J* = 8.4 Hz, 1H), 4.06 – 4.08 (m, 2H), 3.91 (d, *J* = 8.7 Hz, 3H), 3.80 (d, *J* = 8.1 Hz, 3H), 3.54 (d, *J* = 7.5 Hz, 3H), 1.41 – 1.44 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.8, 163.6, 161.4, 160.9, 159.8, 146.2, 143.2, 129.2, 128.4, 121.1, 115.0, 105.9, 63.8, 53.2, 53.1, 52.9, 14.7; HRMS (Q-TOF, m/z) calcd for $C_{19}H_{19}NO_8Na$ $[M+Na]^+$ 412.1008, found 412.1015.

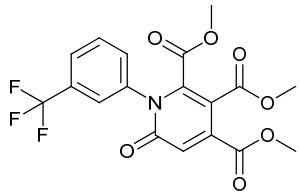


Triethyl 6-oxo-1-p-tolyl-1,6-dihydropyridine-2,3,4-tricarboxylate (5ai): oil; 126.7 mg; 63% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.18-7.20 (d, *J* = 8.0 Hz, 2H), 7.04-7.06 (d, *J* = 8.0 Hz, 2H), 6.75 (s, 1H), 4.26-4.32 (q, *J* = 7.1 Hz, 2H), 4.15-4.21 (q, *J* = 7.1 Hz, 2H), 3.86-3.91 (d, *J* = 21.3 Hz, 2H), 2.31 (s, 3H), 1.27-1.29 (t, *J* = 7.1 Hz, 3H), 1.19-1.21 (d, *J* = 7.2 Hz, 3H), 0.88-0.92 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.2, 163.2, 160.8, 145.7, 143.6, 140.0, 133.7, 129.9, 128.0, 121.1, 106.8, 62.6, 62.3, 62.0, 29.6, 21.2, 13.9, 13.8, 13.3; HRMS (Q-TOF, m/z) calcd for $C_{21}H_{23}NO_7Na$

$[M+Na]^+$ 424.1372, found 424.1378.



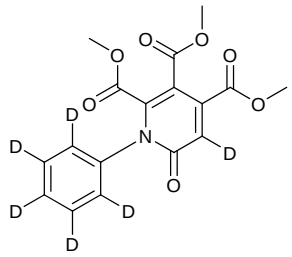
Trimethyl 1-(4-nitrophenyl)-6-oxo-1,6-dihydropyridine-2,3,4-tricarboxylate (5aj): white solid; 96.2 mg; 49% yield; mp 145–149 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.34-8.36 (d, J = 8.8 Hz, 2H), 7.46-7.48 (d, J = 8.8 Hz, 2H), 6.88 (s, 1H), 3.92 (s, 3H), 3.82 (s, 3H), 3.56 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.3, 163.5, 161.1, 160.1, 148.5, 144.2, 143.4, 141.8, 129.7, 124.8, 122.1, 108.4, 53.6, 53.4, 53.3; HRMS (Q-TOF, m/z) calcd for $C_{17}H_{14}N_2O_9Na$ $[M+Na]^+$, 413.0597, found. 413.0591



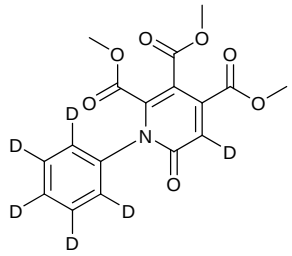
Trimethyl 6-oxo-1-(3-(trifluoromethyl)phenyl)-1,6-dihydropyridine-2,3,4-tricarboxylate (5ak): white solid; 80.5 mg; 39% yield; mp 92–94 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (d, *J* = 7.9 Hz, 1H), 7.64 (t, *J* = 7.9 Hz, 1H), 7.54 (s, 1H), 7.50 (d, *J* = 8.1 Hz, 1H), 6.86 (s, 1H), 3.93 (s, 3H), 3.81 (s, 3H), 3.52 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.5, 163.5, 161.2, 160.4, 145.1, 143.5, 137.0, 132.1, 130.2, 129.9, 125.4, 125.1, 121.8, 107.6, 53.4, 53.3, 53.2; ^{19}F NMR (377 MHz, $CDCl_3$) δ -63.2; HRMS (Q-TOF, m/z) calcd for $C_{18}H_{14}F_3NO_7Na$ $[M+Na]^+$ 436.0620, found 436.0607.

6-1 and 6-2: white solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.67 (s, 1H), 7.27 (s, 2H), 7.08 (s, 1H), 6.90 (d, *J* = 6.6 Hz, 2H), 5.39 (s, 1H), 3.73 (s, 3H), 3.68 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.9, 164.9, 148.0, 140.3, 129.2, 124.3, 120.7, 93.6, 52.8,

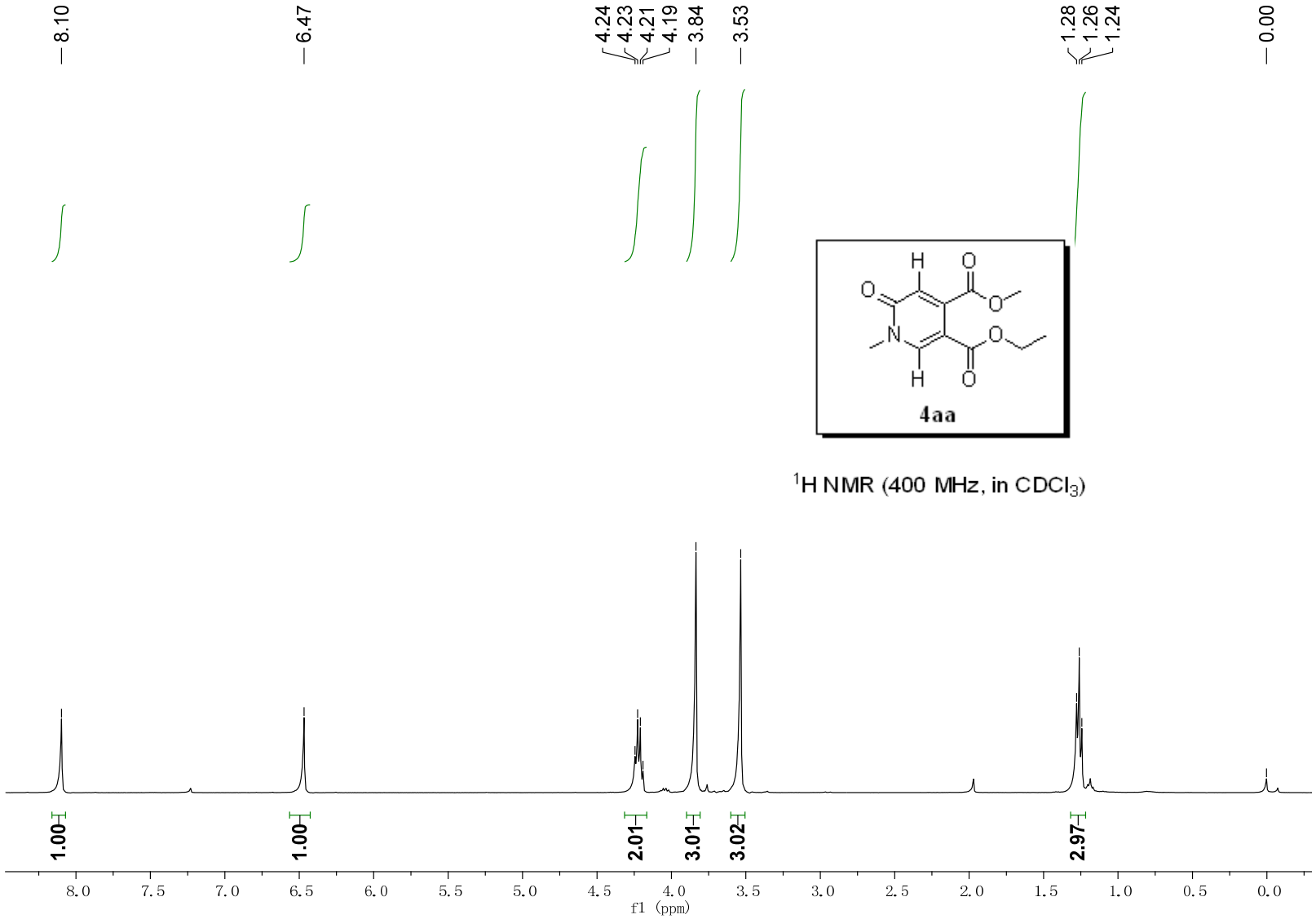
51.3.

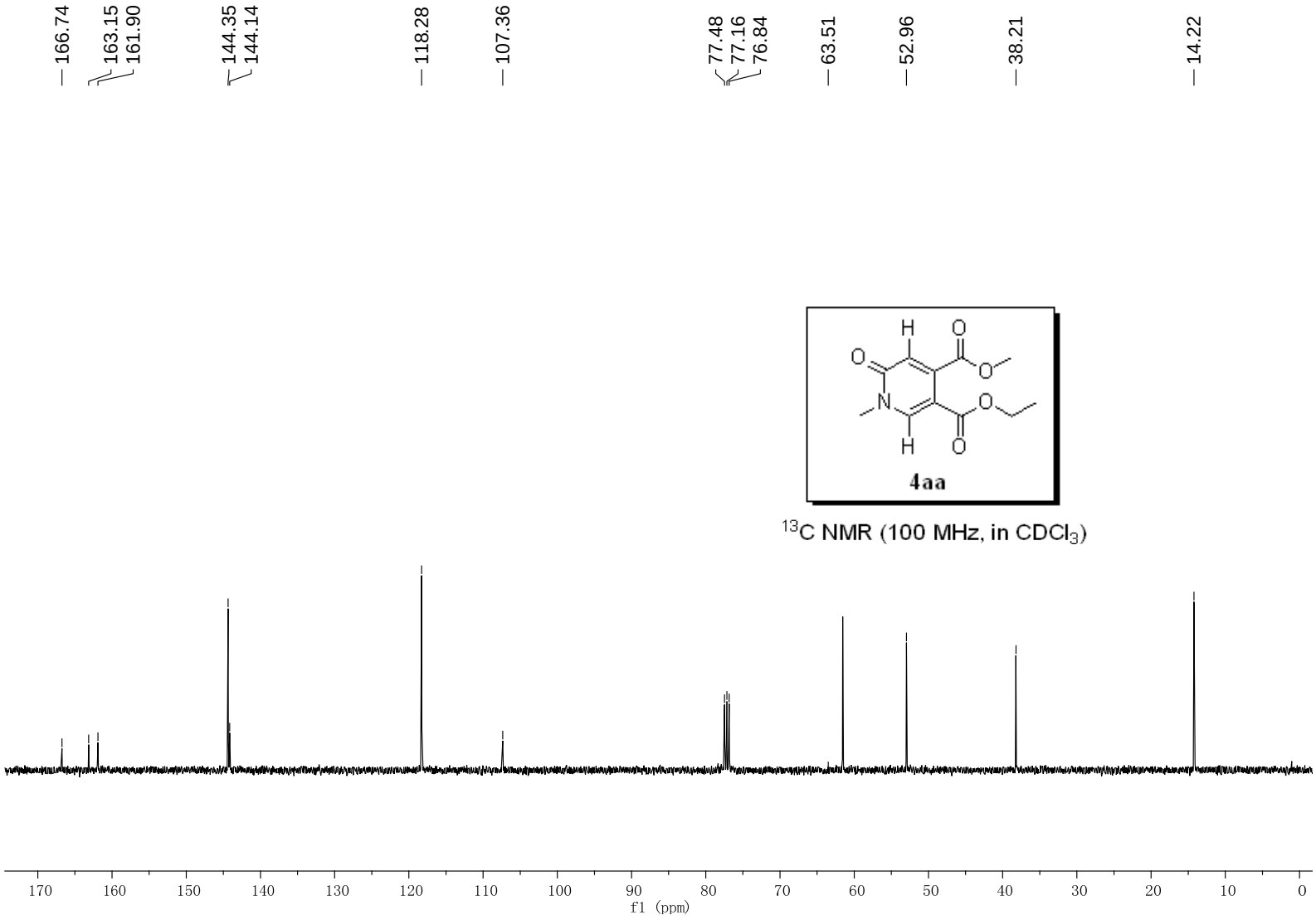


D-Trimethyl 6-oxo-1-phenyl-1,6-dihydropyridine-2,3,4-tricarboxylate (D-5aa): white solid; 137.1 mg; 79% yield; mp 115–117 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 (s, 3.00H), 7.25 (d, J = 4.4 Hz, 2.00H), 6.83 (s, 0.48H), 3.91 (s, 3.00H), 3.80 (s, 3.00H), 3.48 (s, 3.00H).



D'-Trimethyl 6-oxo-1-phenyl-1,6-dihydropyridine-2,3,4-tricarboxylate (D'-5aa): white solid; 180 mg (1 mmol); 51% yield; mp 121 – 122 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.83 (s, 0.17H), 3.92 (s, 2.99H), 3.80 (s, 3.02H), 3.48 (s, 3H); HRMS (Q-TOF, m/z) calcd for $C_{17}H_9D_6NO_7Na$ $[M+Na]^+$ 374.1123, found 374.1112.

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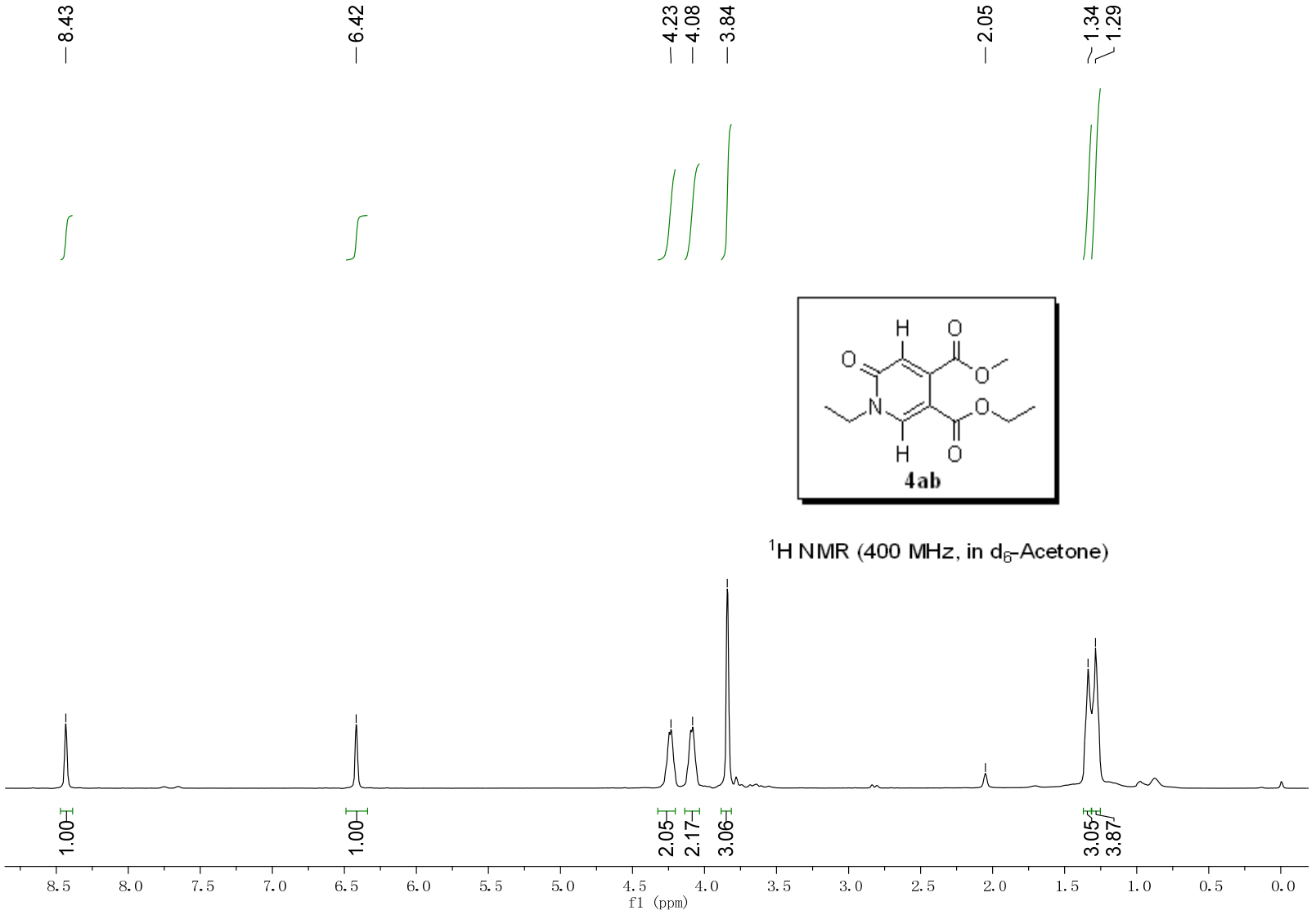
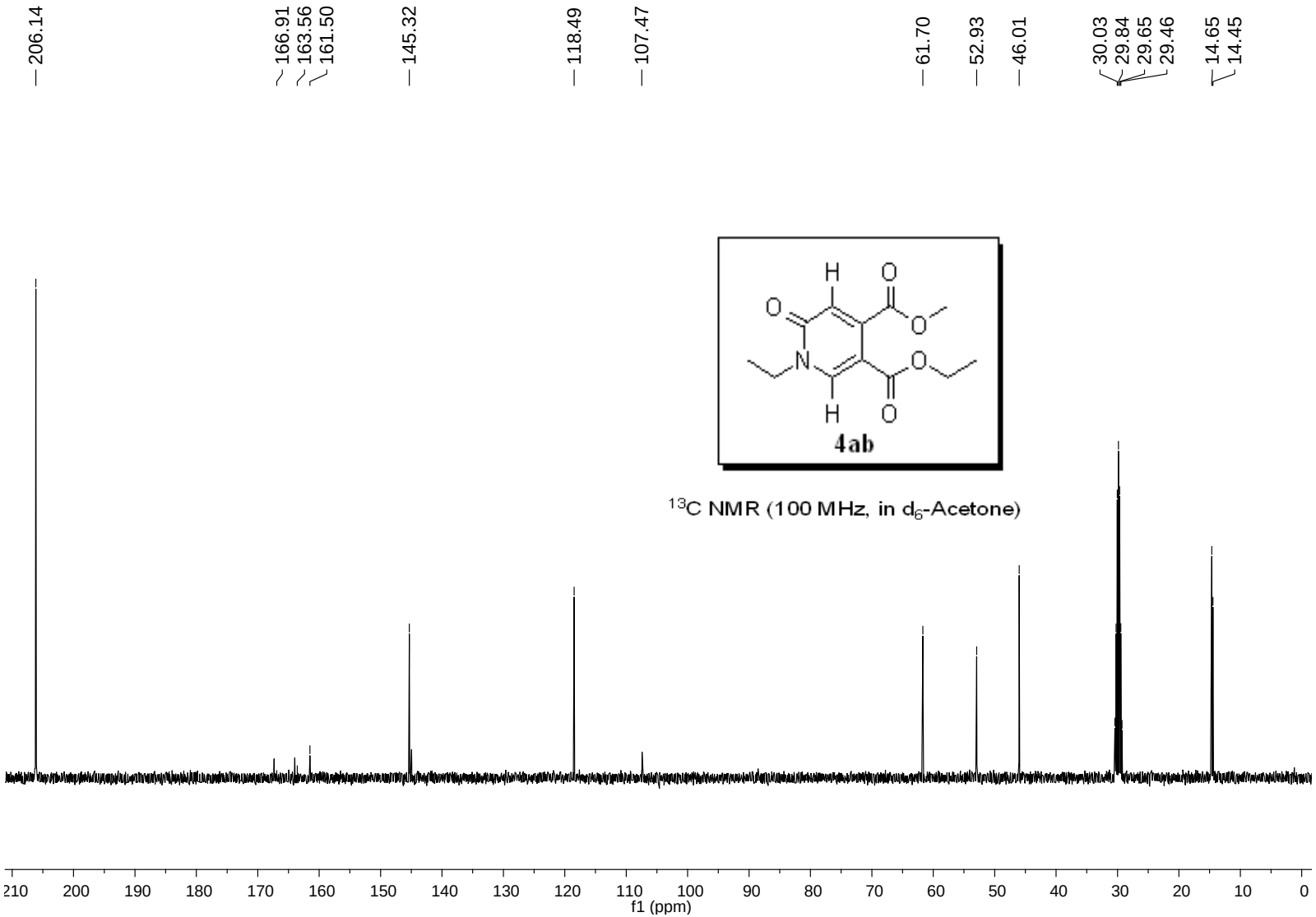
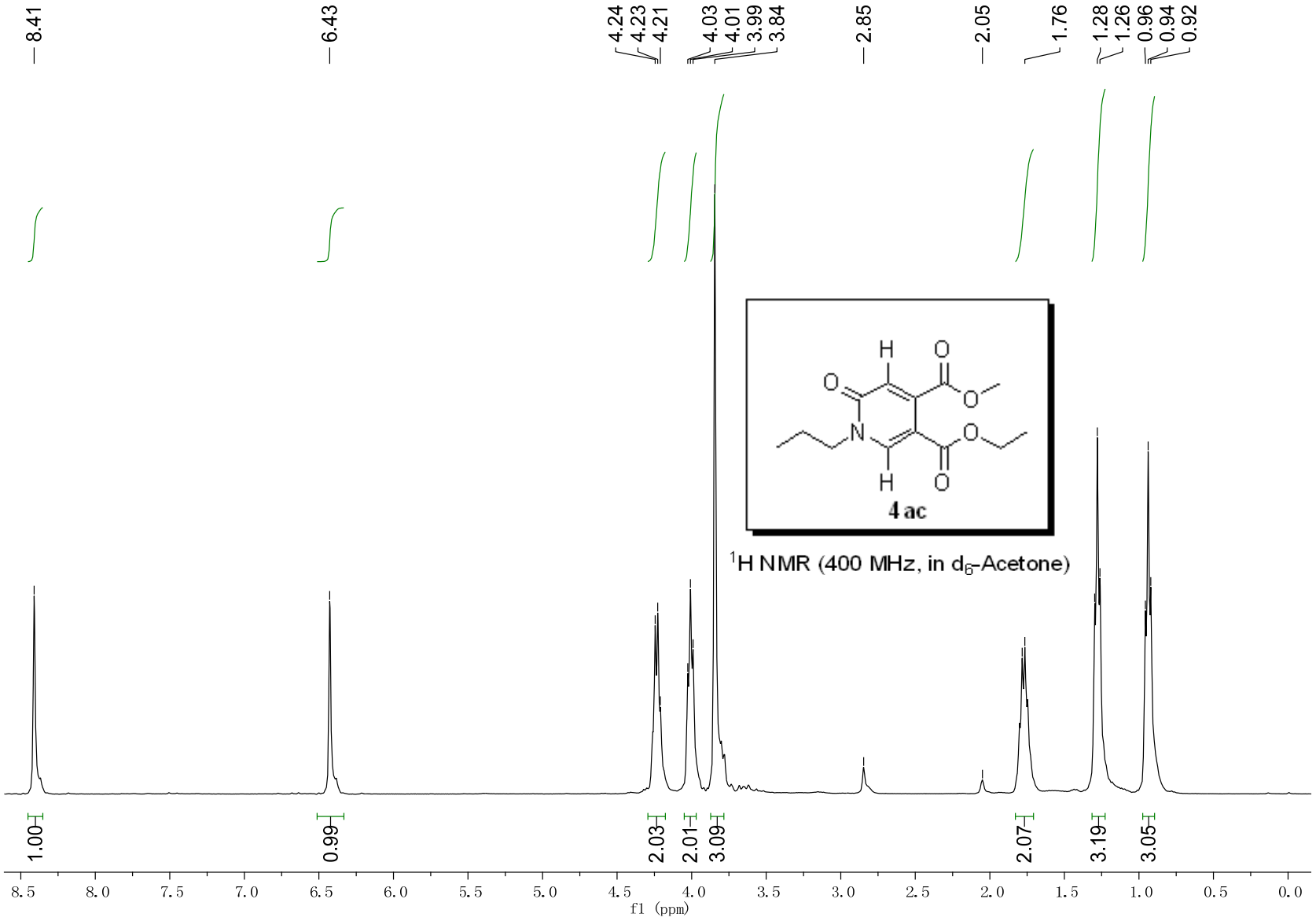
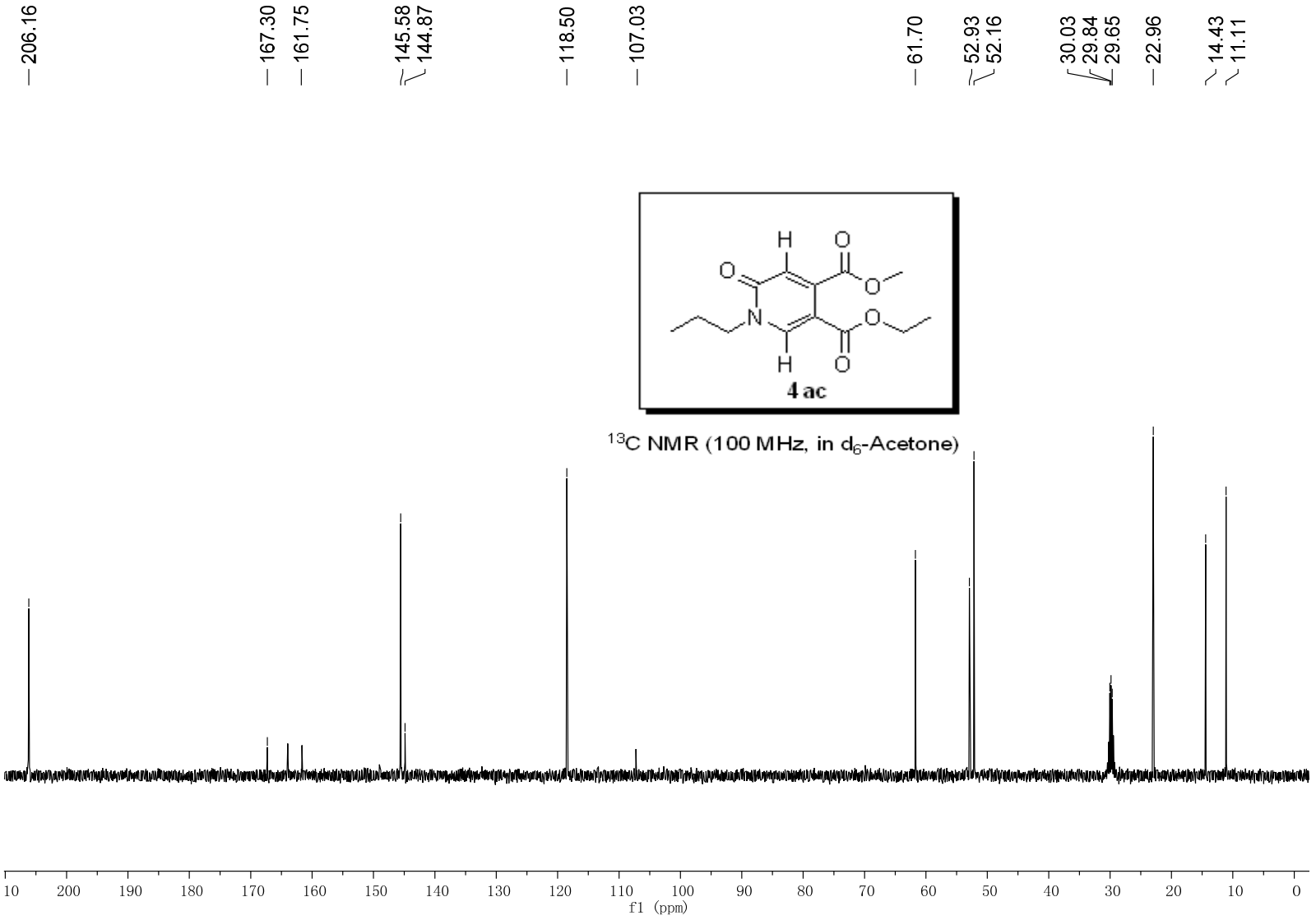
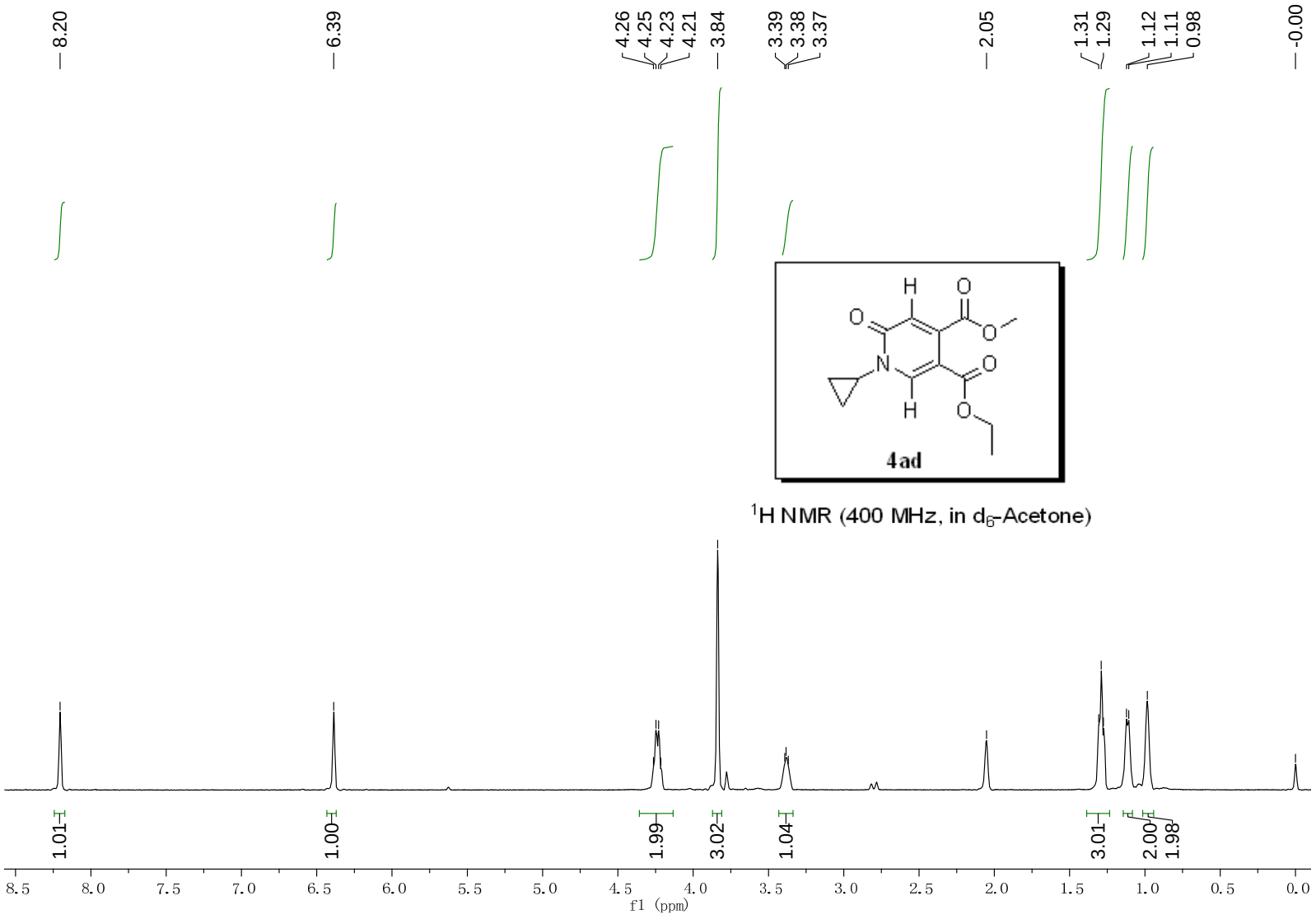
163.15
161.90
144.35
144.14
118.28
107.36
77.48
77.16
76.84
63.51
52.96
38.21
14.22
4aa
13C NMR (100 MHz, in CDCl3)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

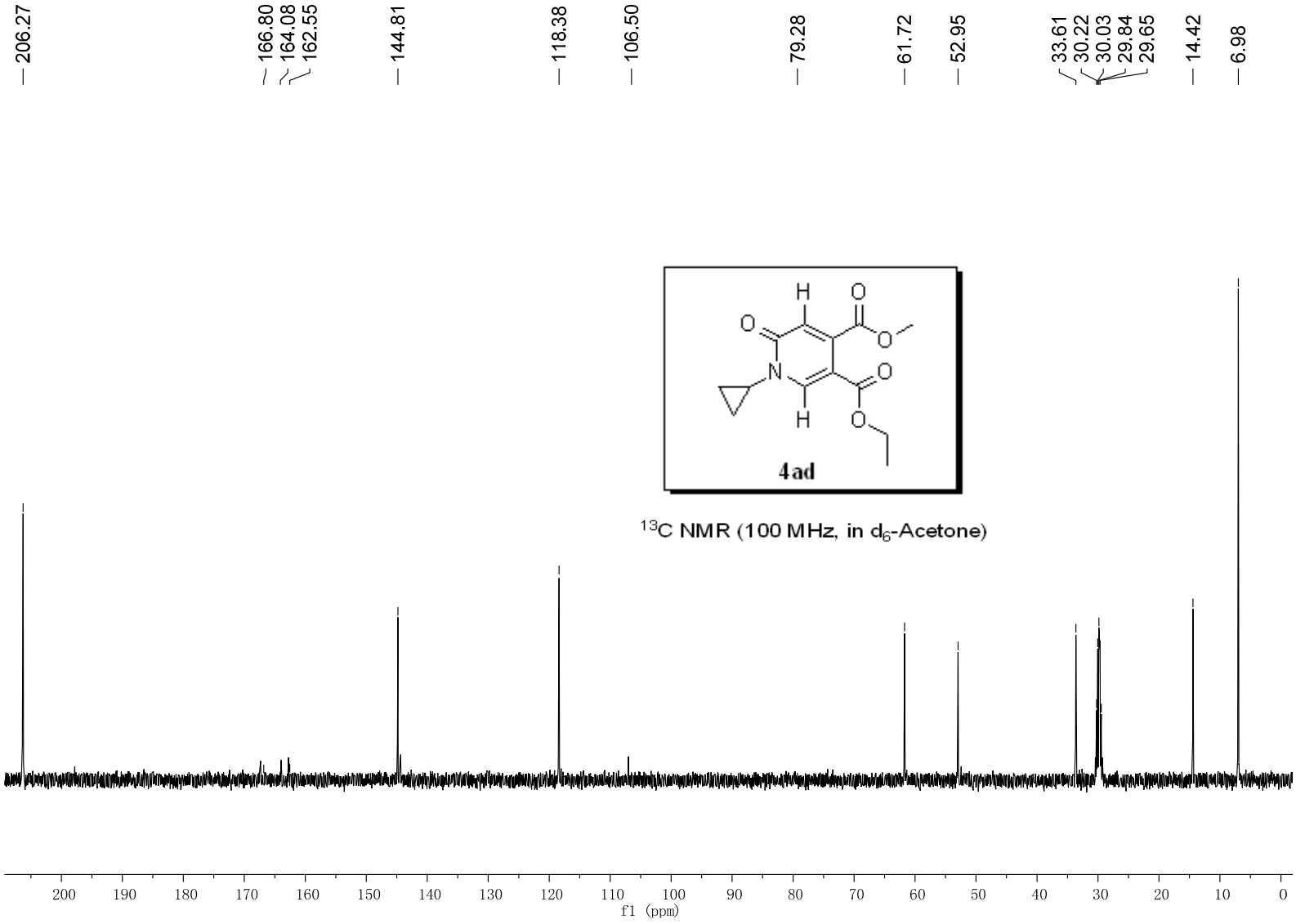


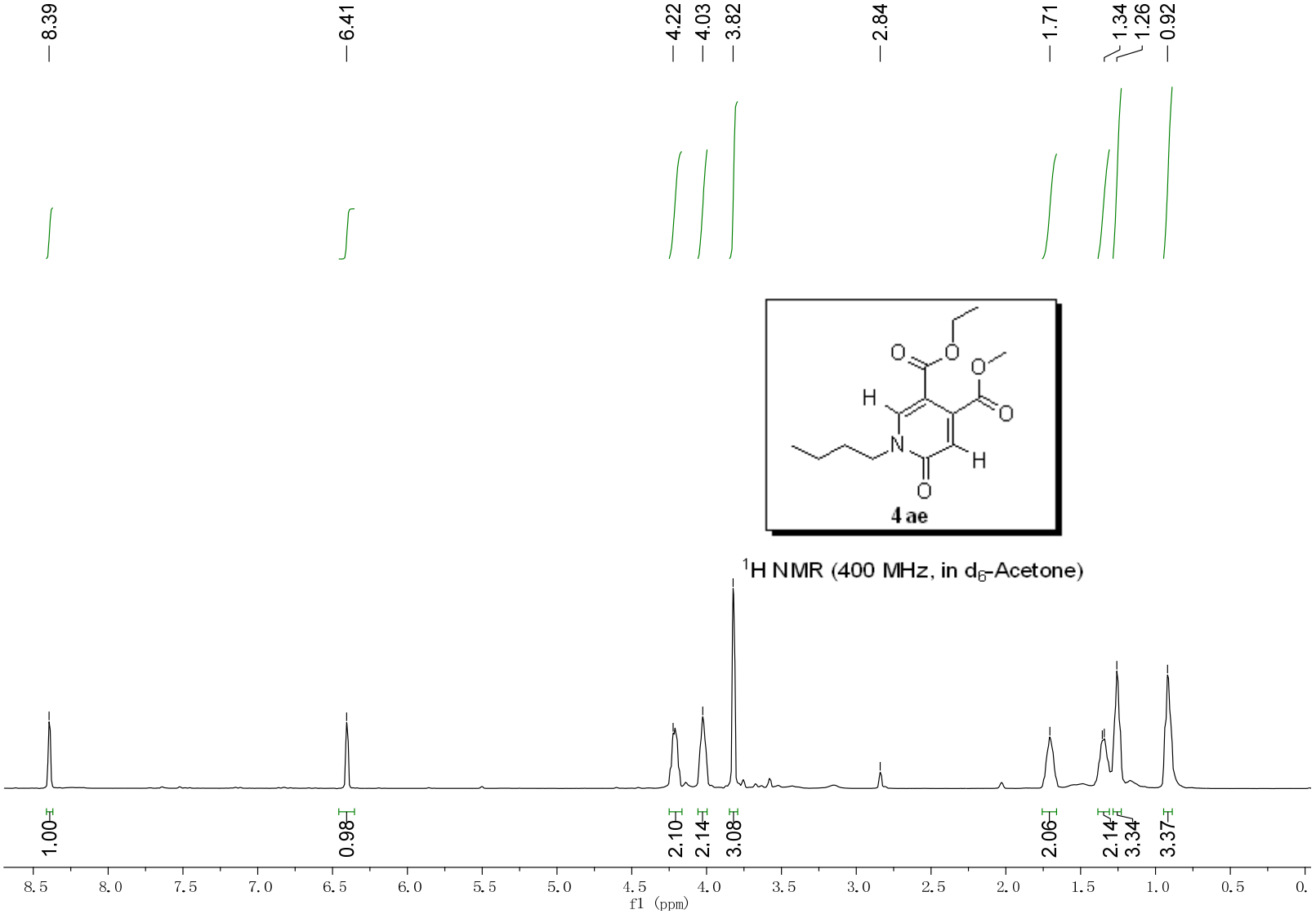


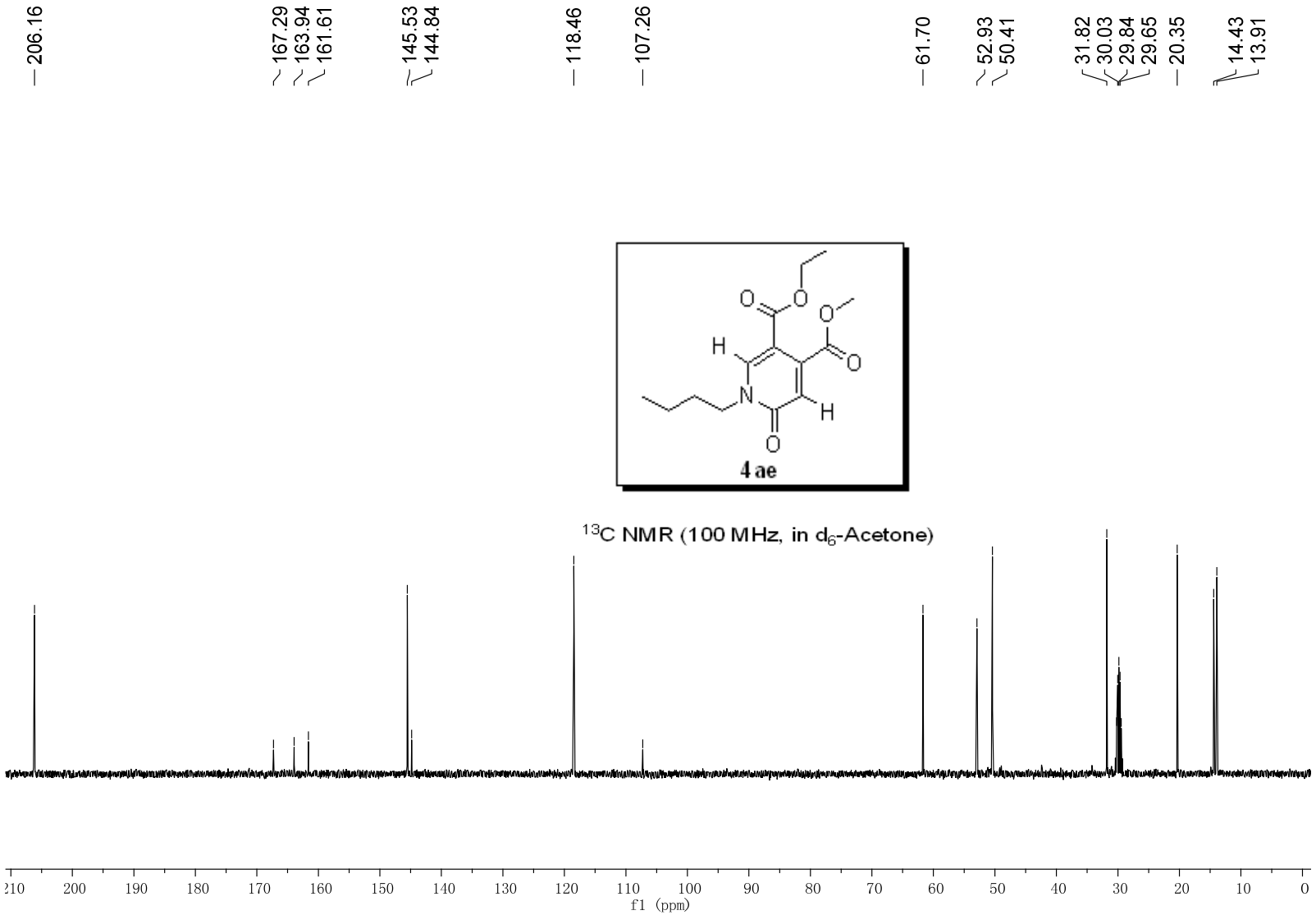


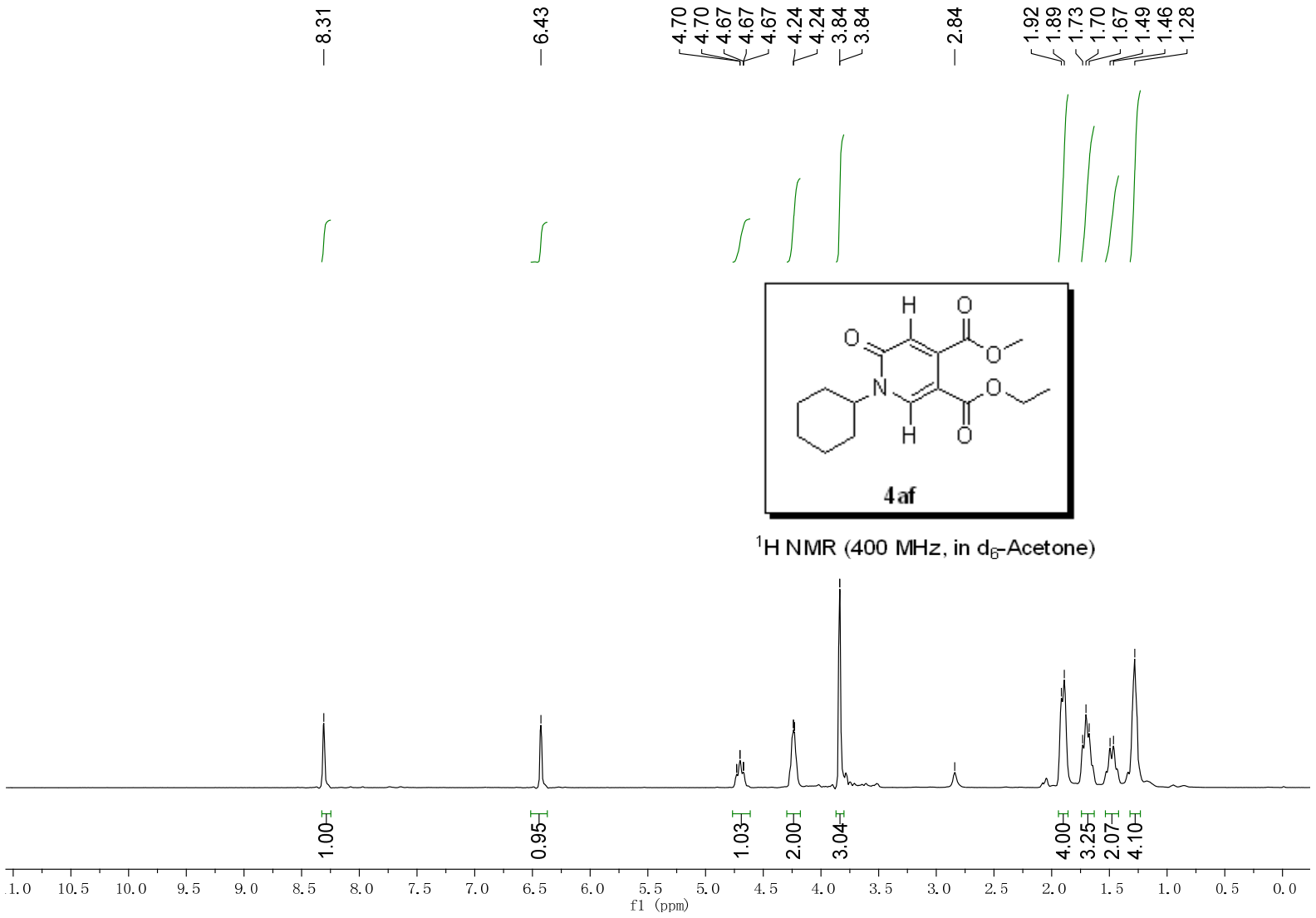


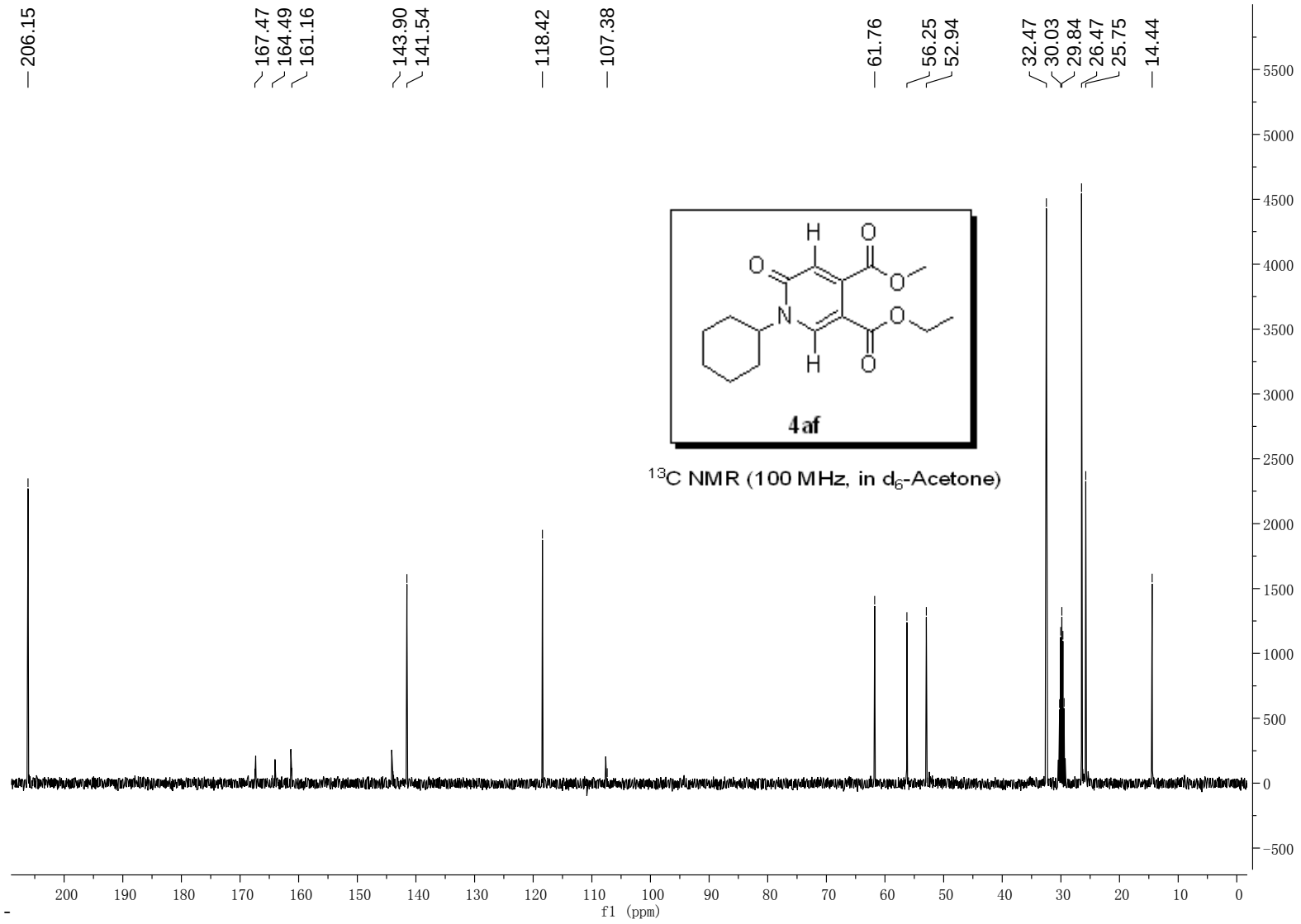


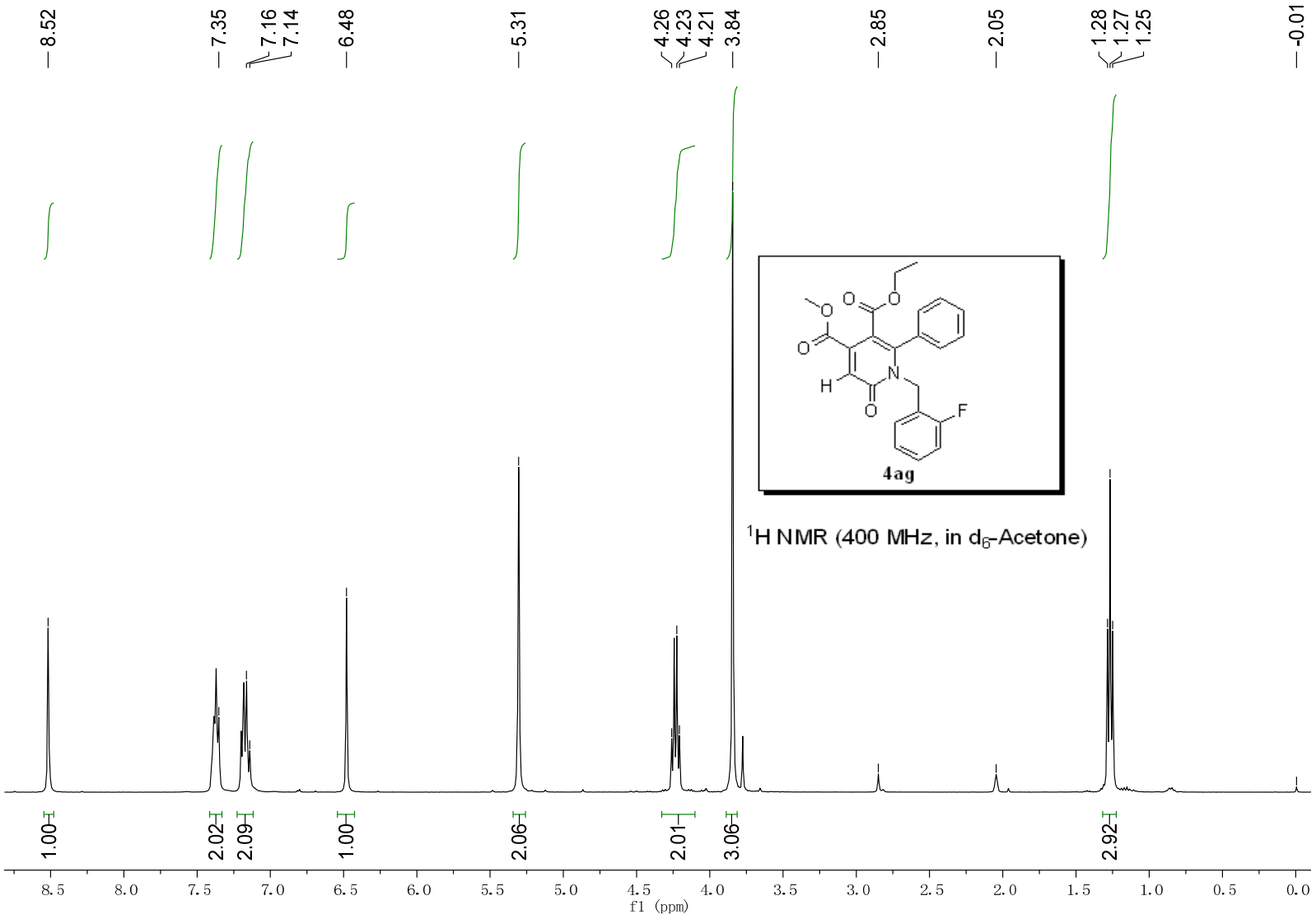


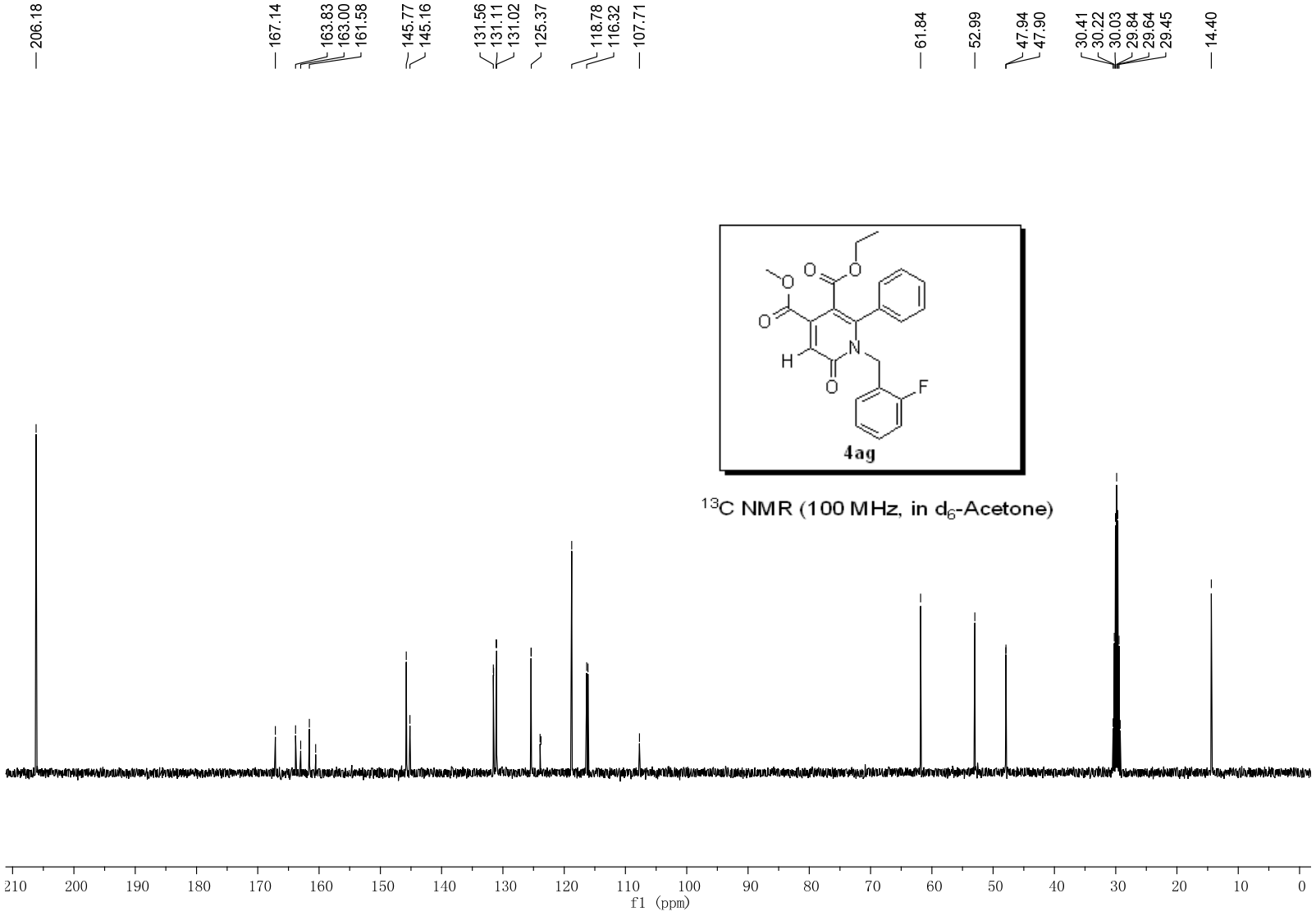


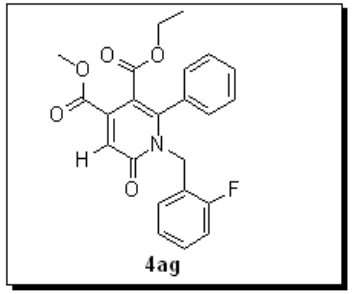






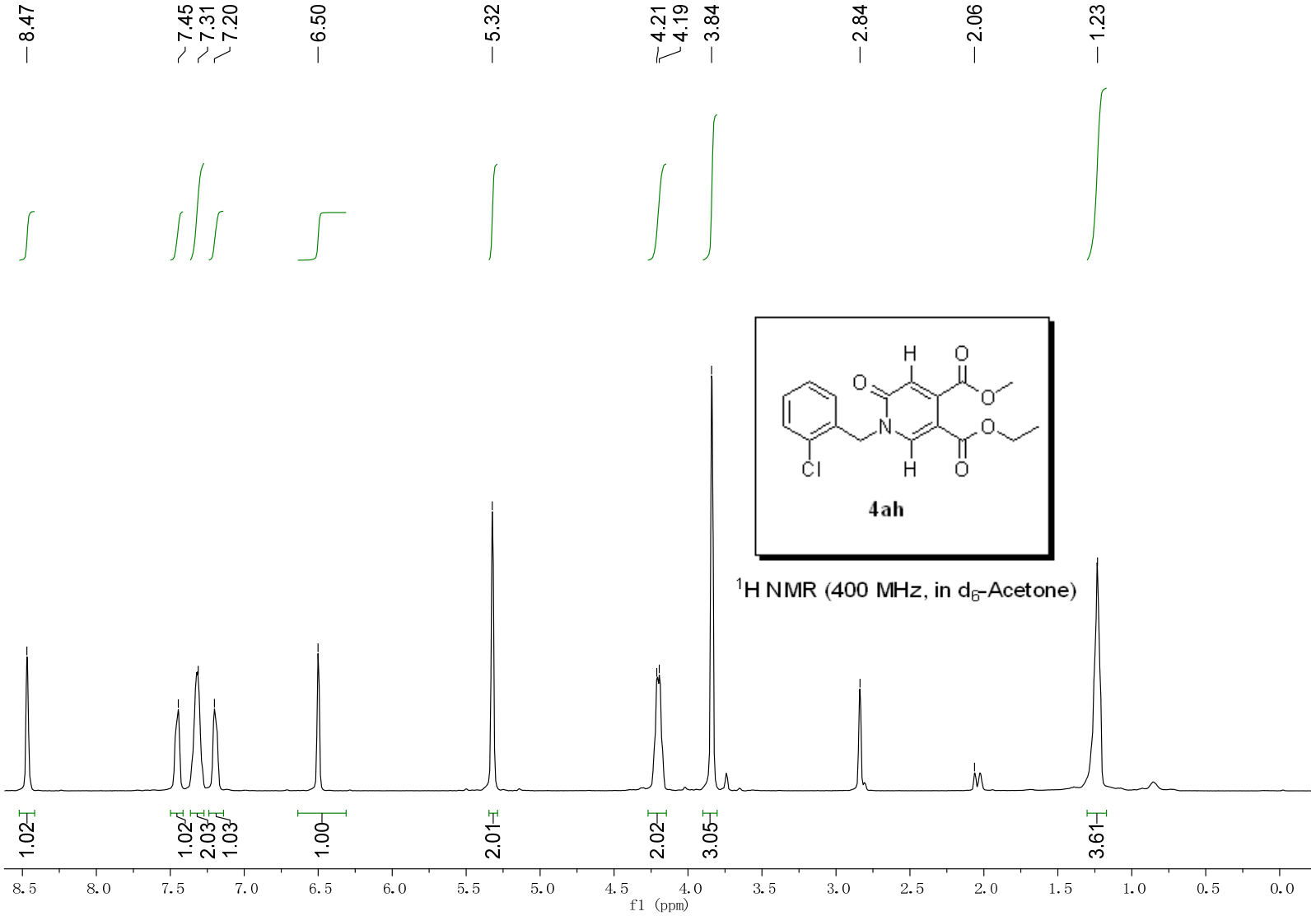


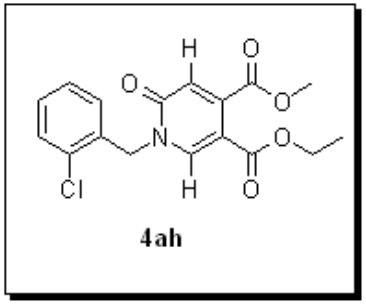




4ag

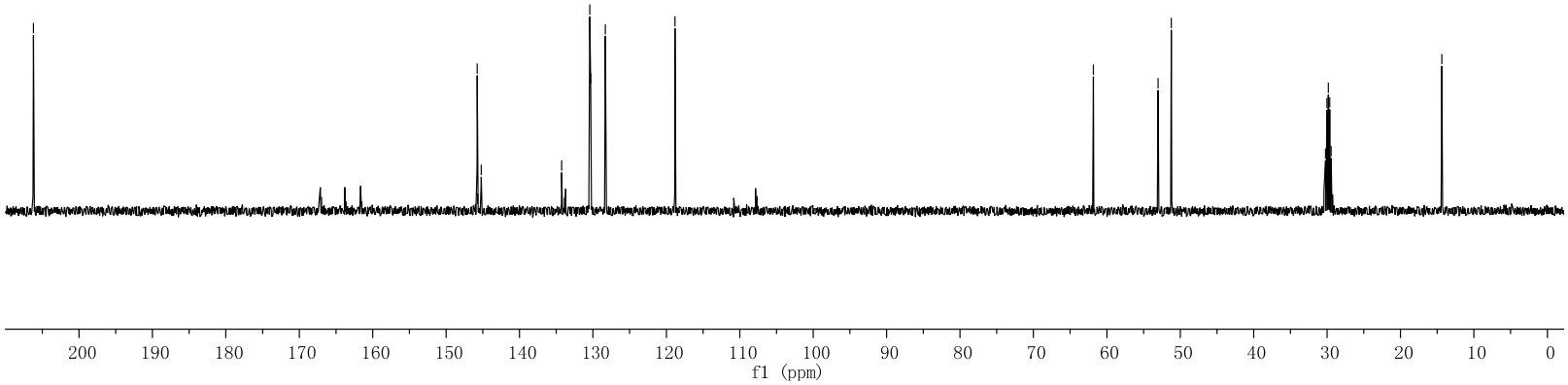
^{19}F NMR (377 MHz, in d_6-Acetone)

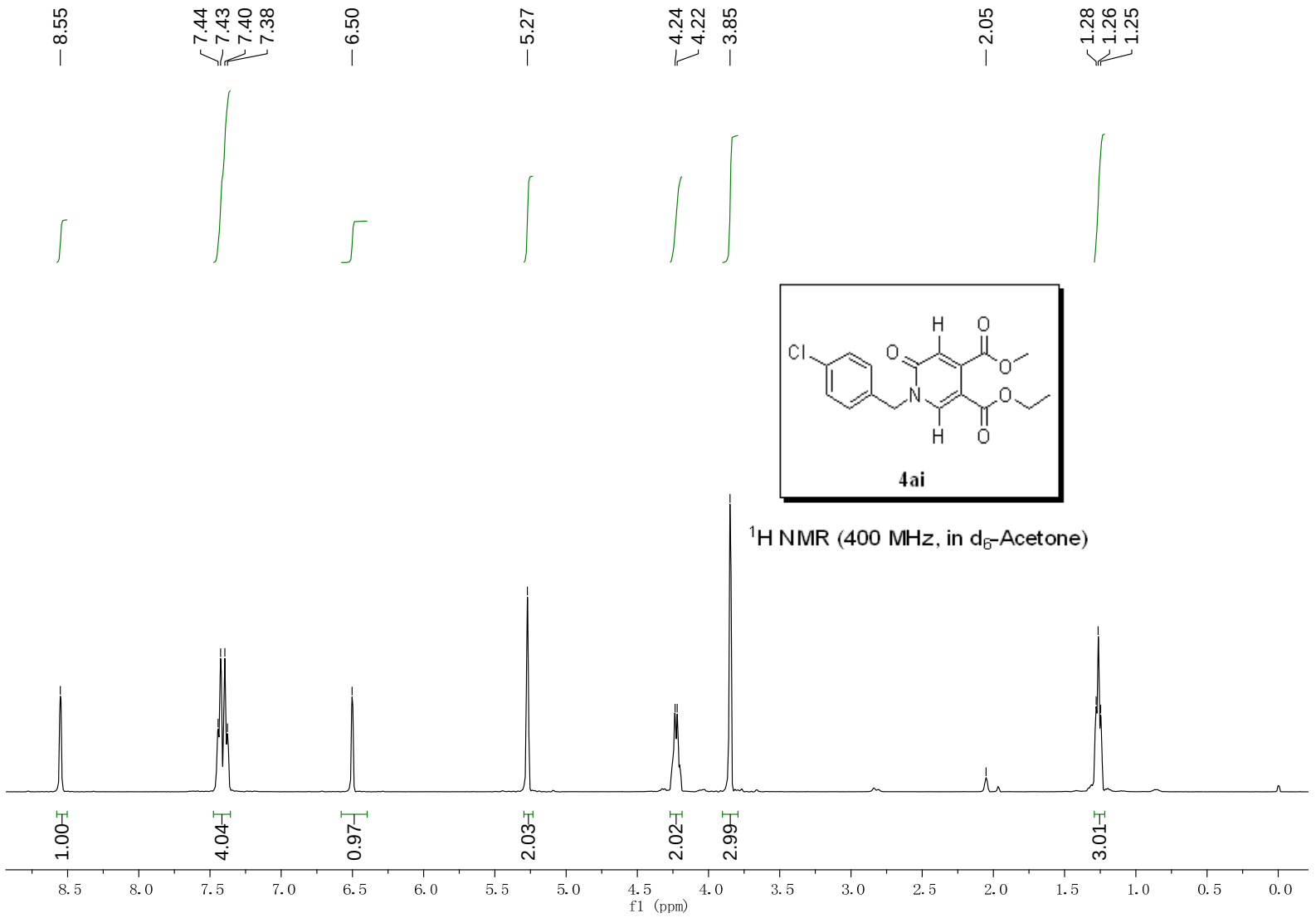


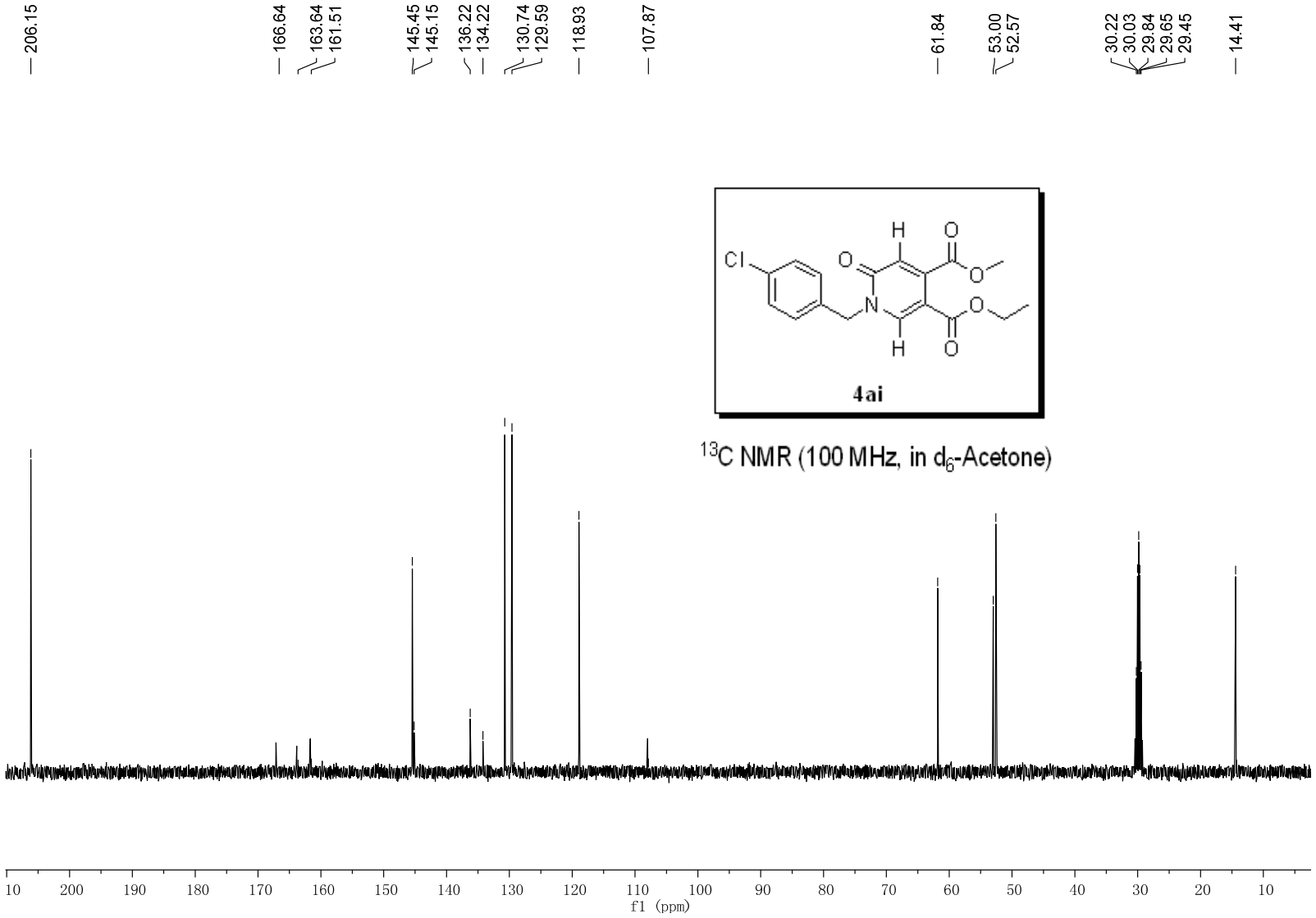


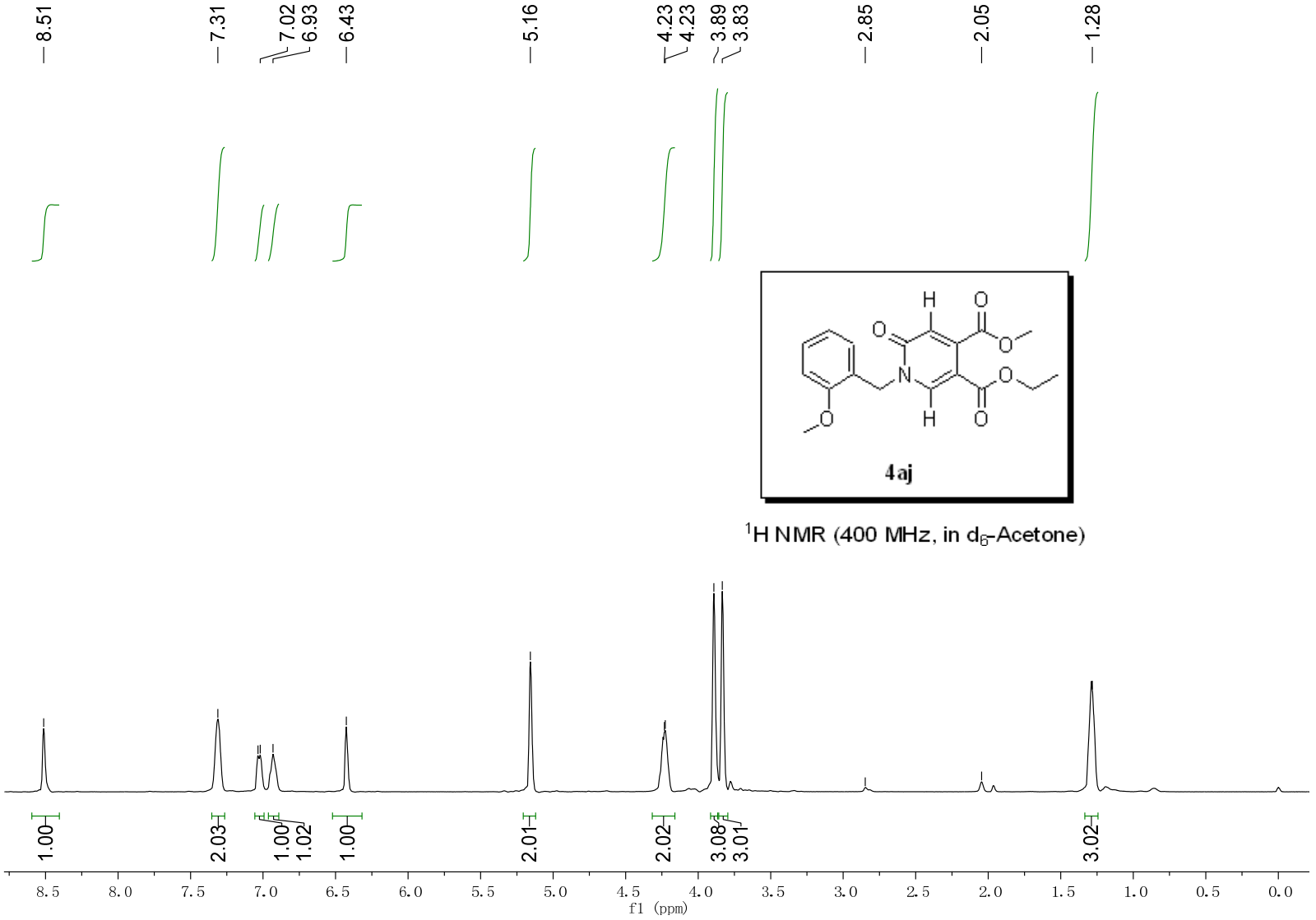
4ah

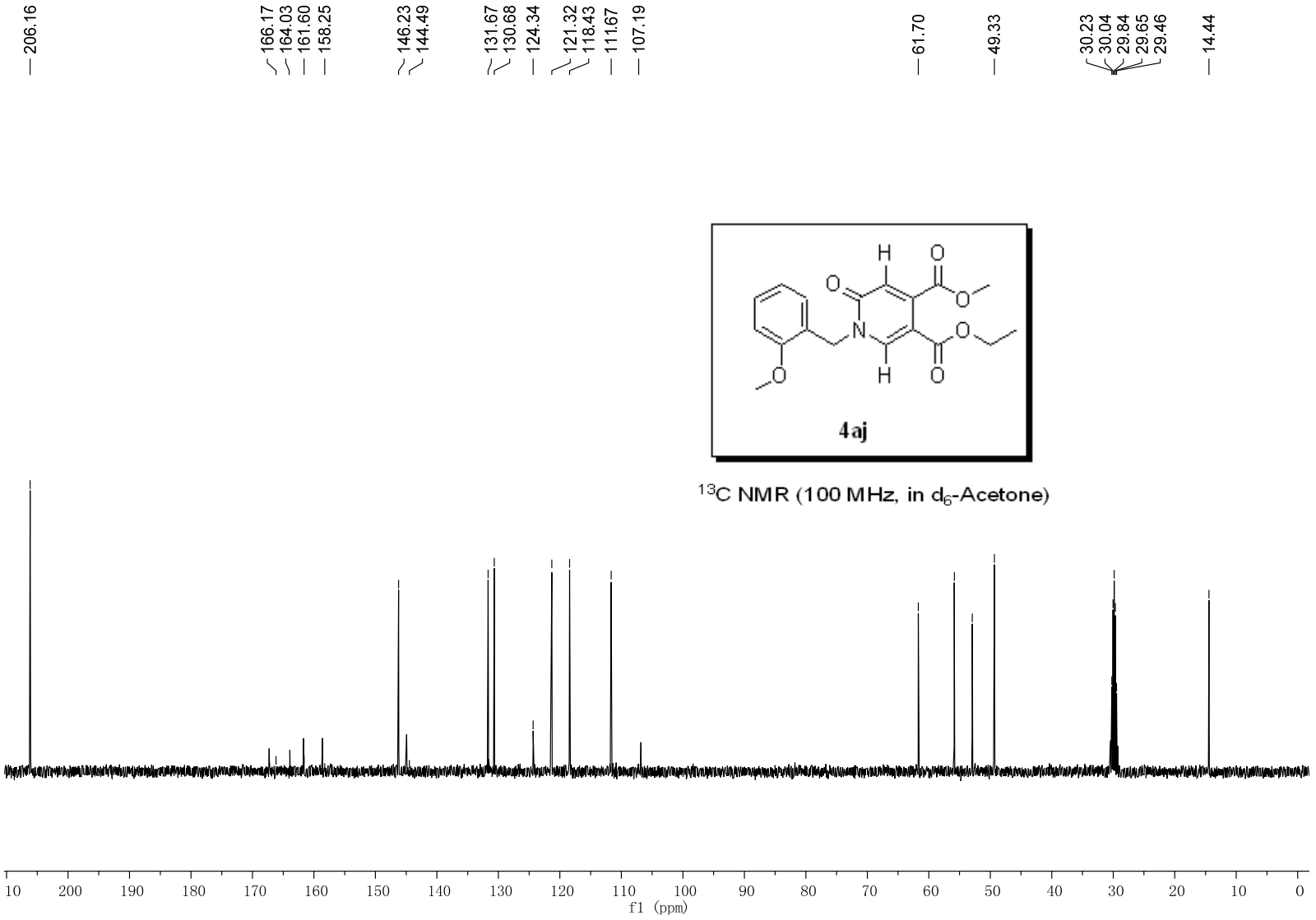
^{13}C NMR (100 MHz, in d_6-Acetone)

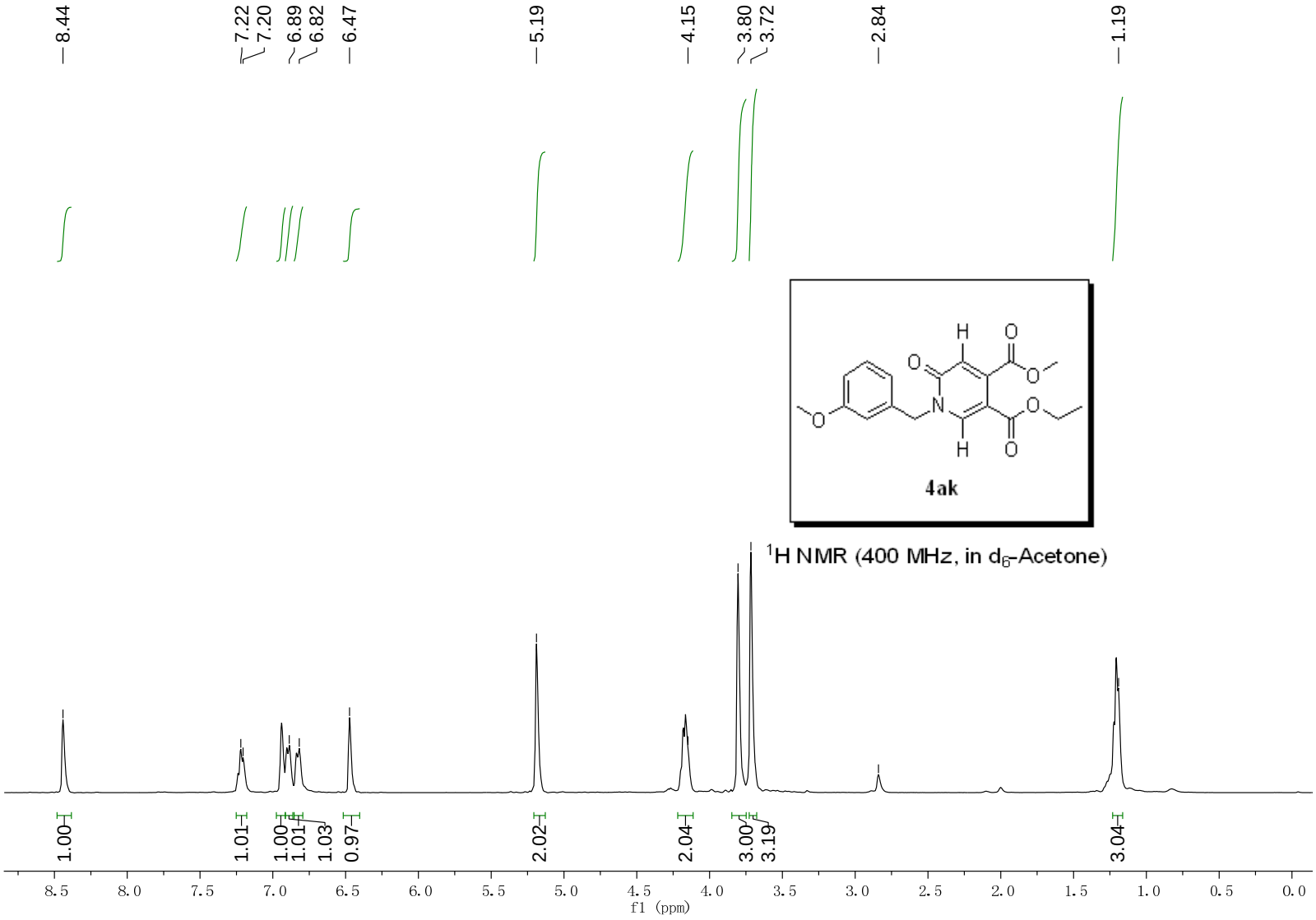












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167.17
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138.72
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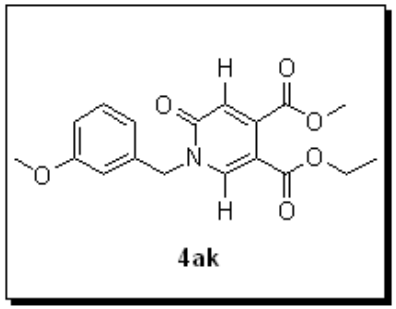
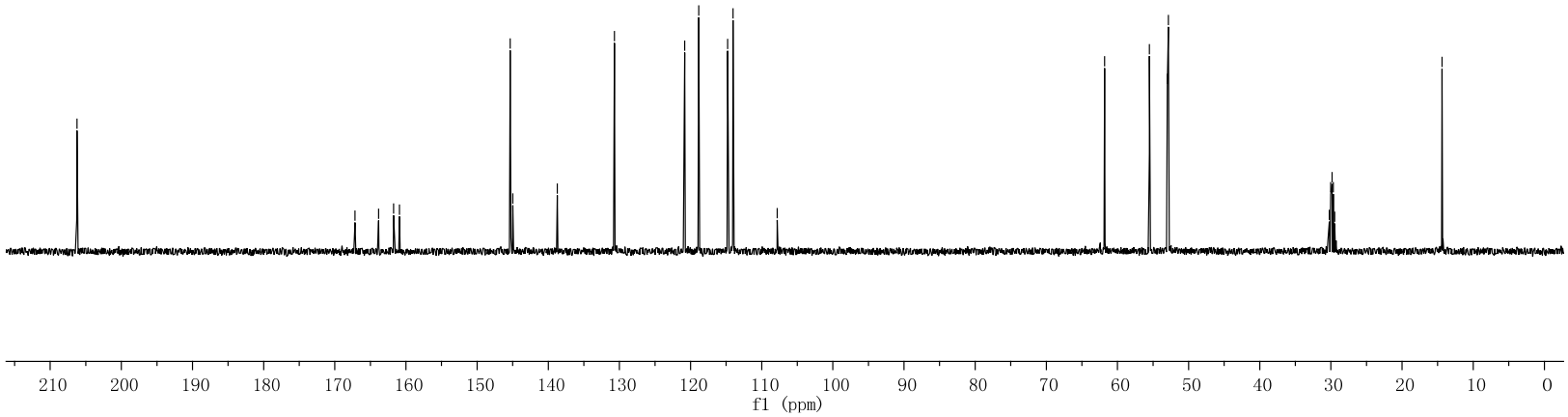
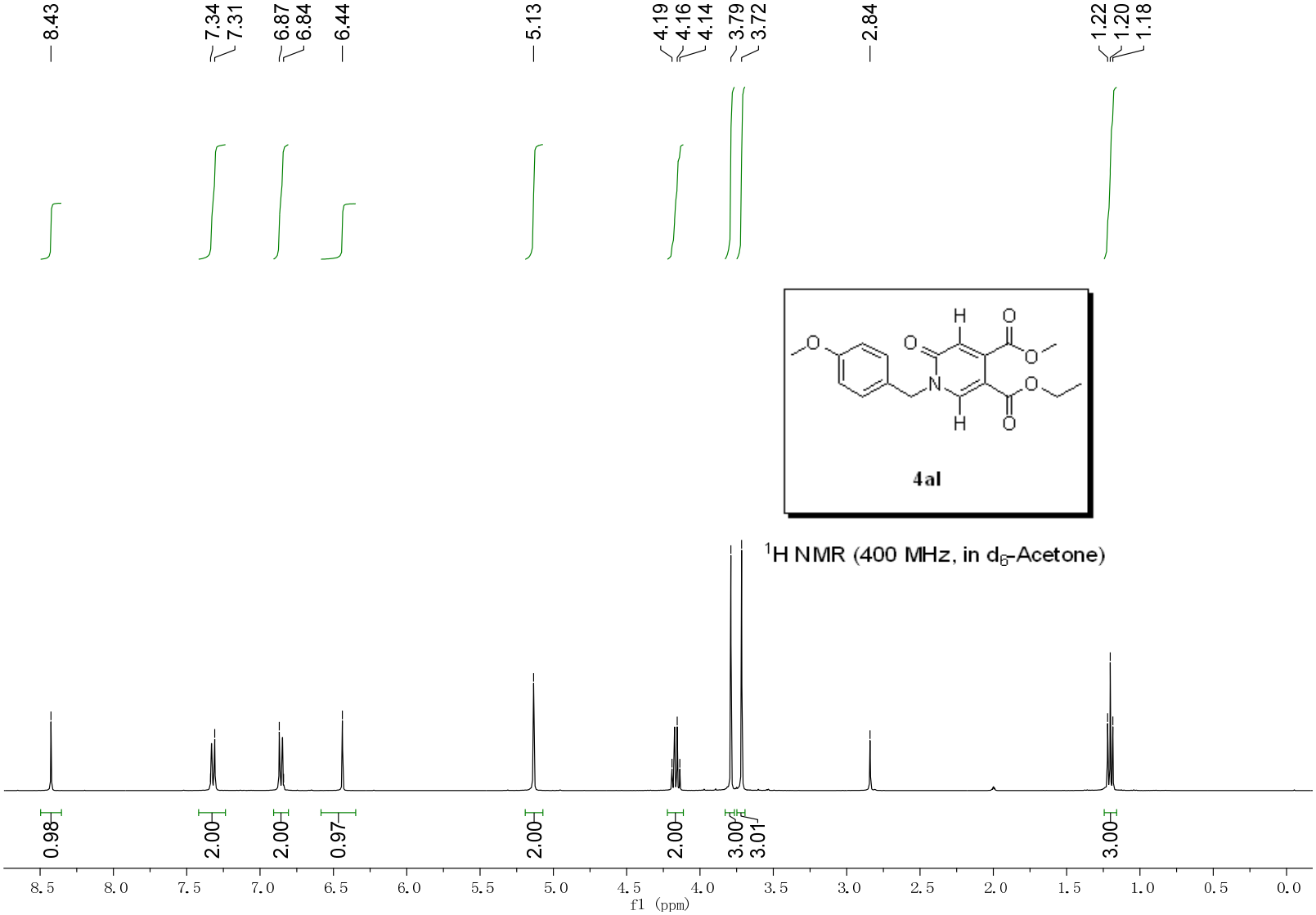
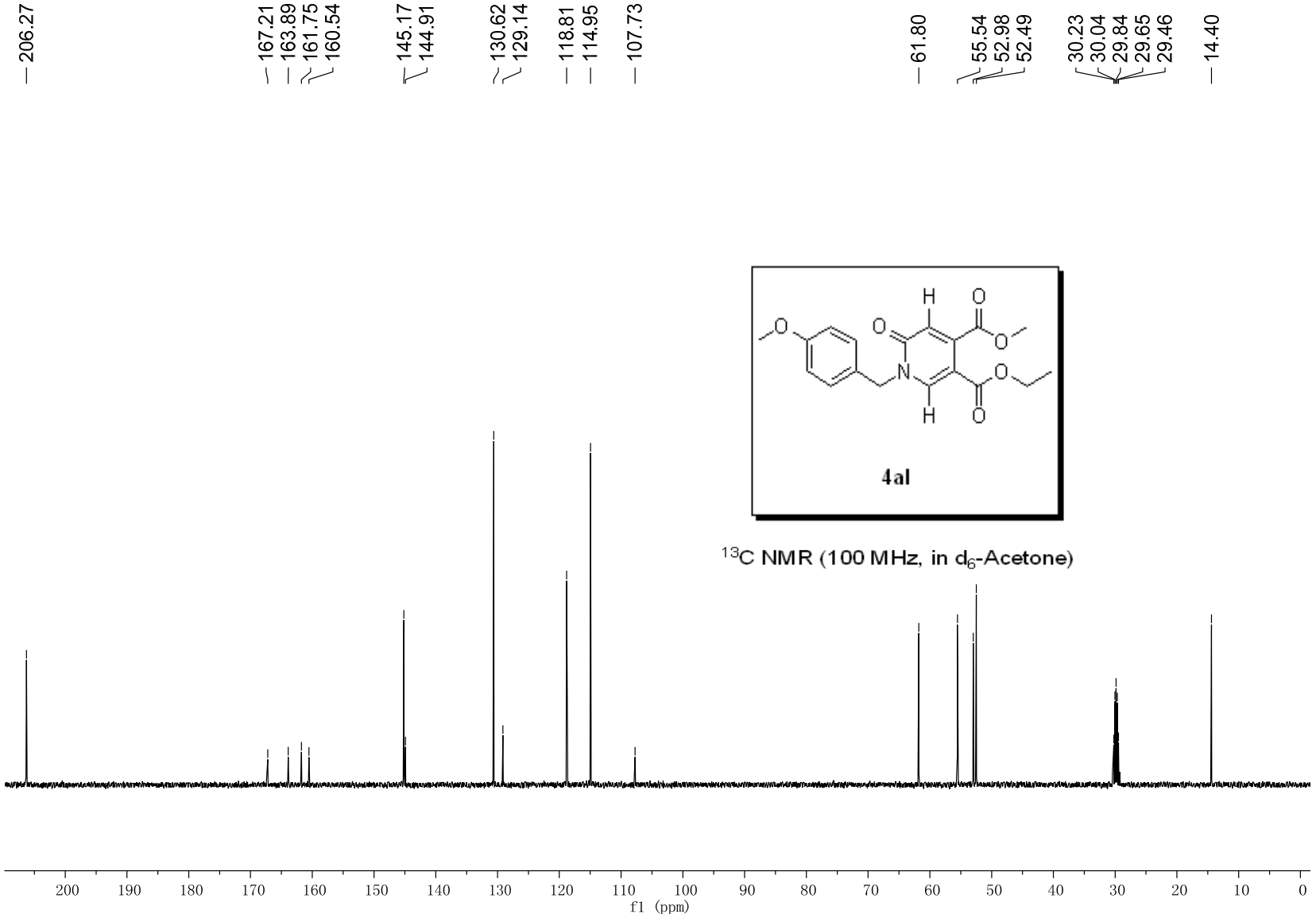
14.39

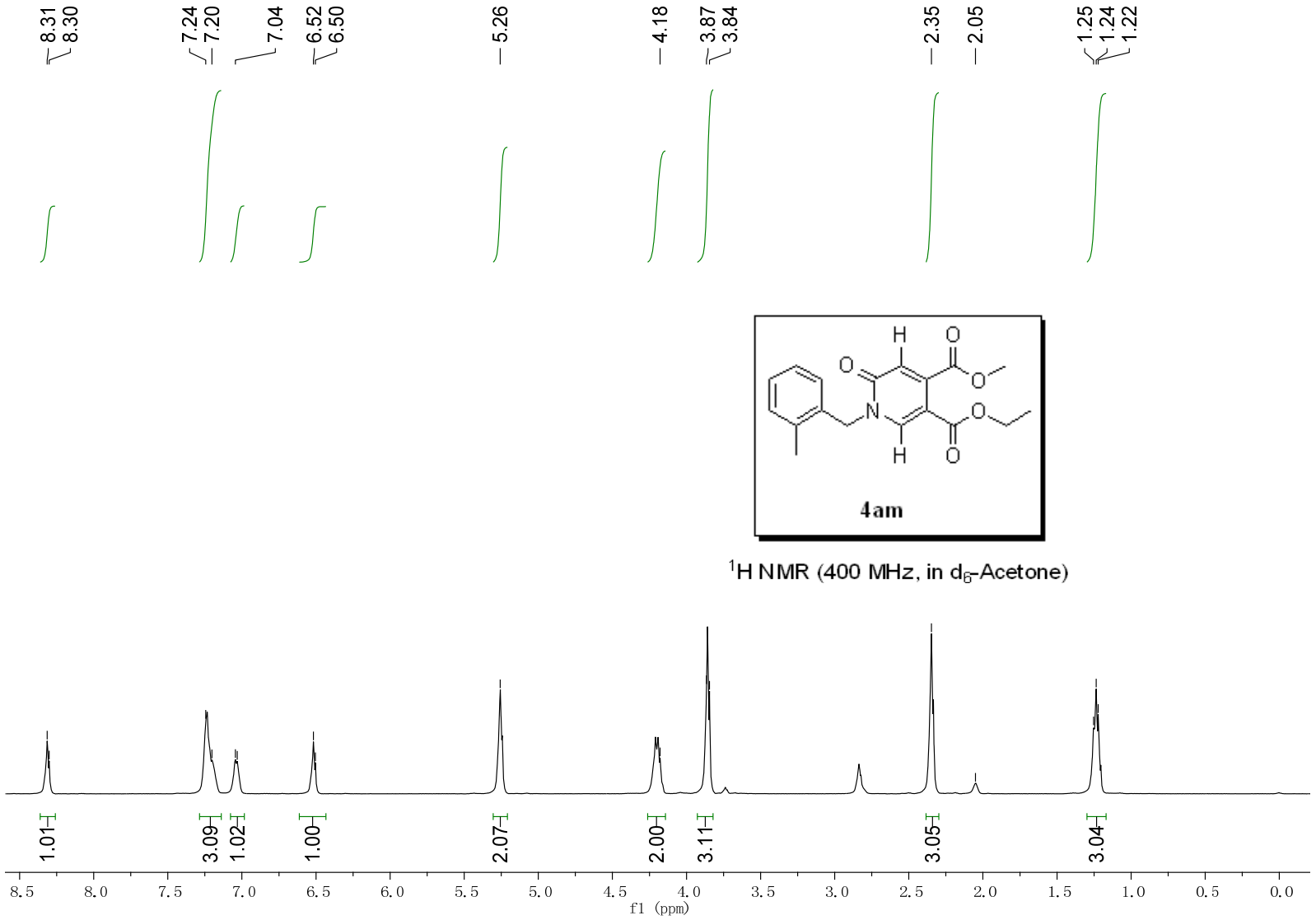


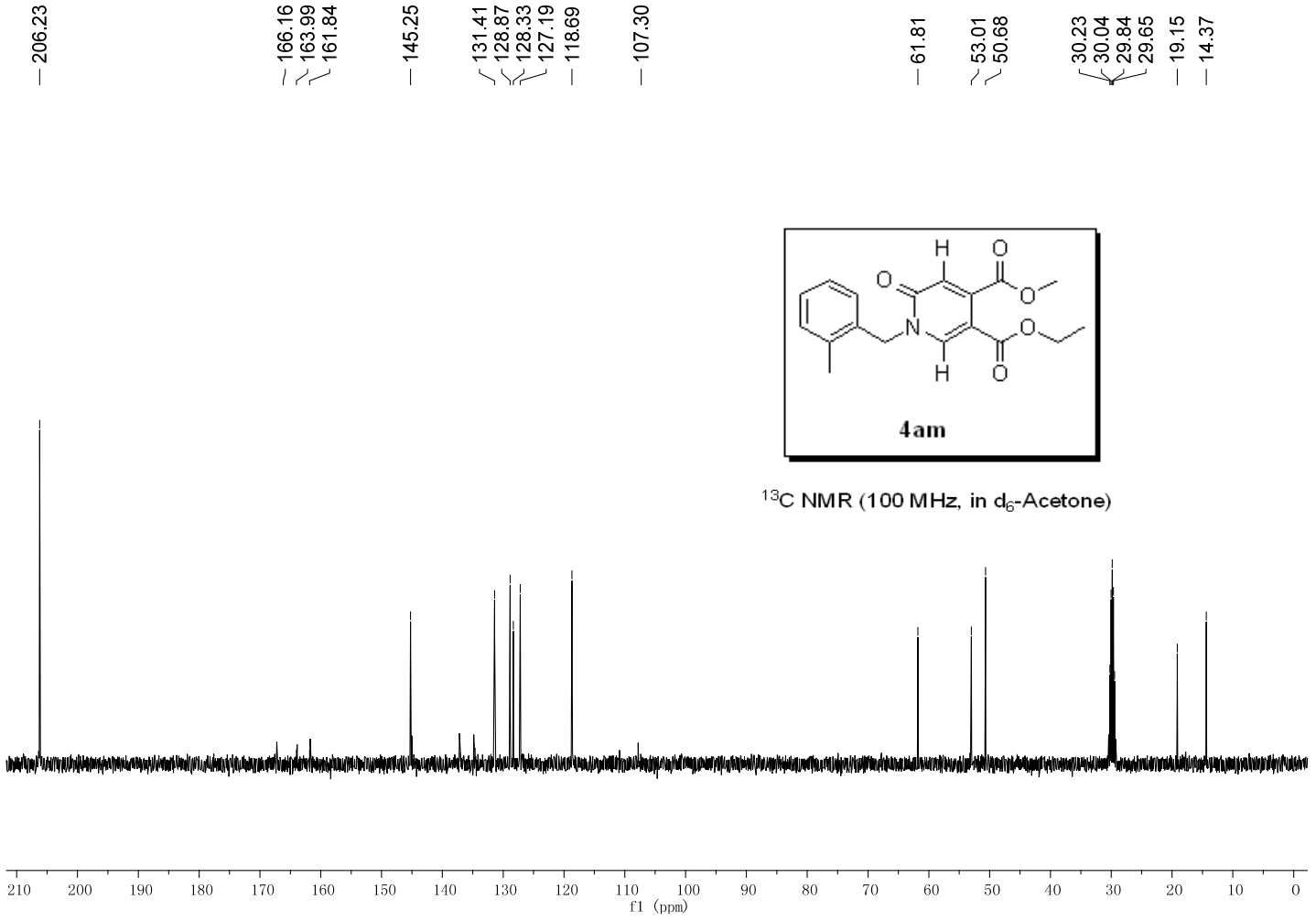
^{13}C NMR (100 MHz, in d_6-Acetone)

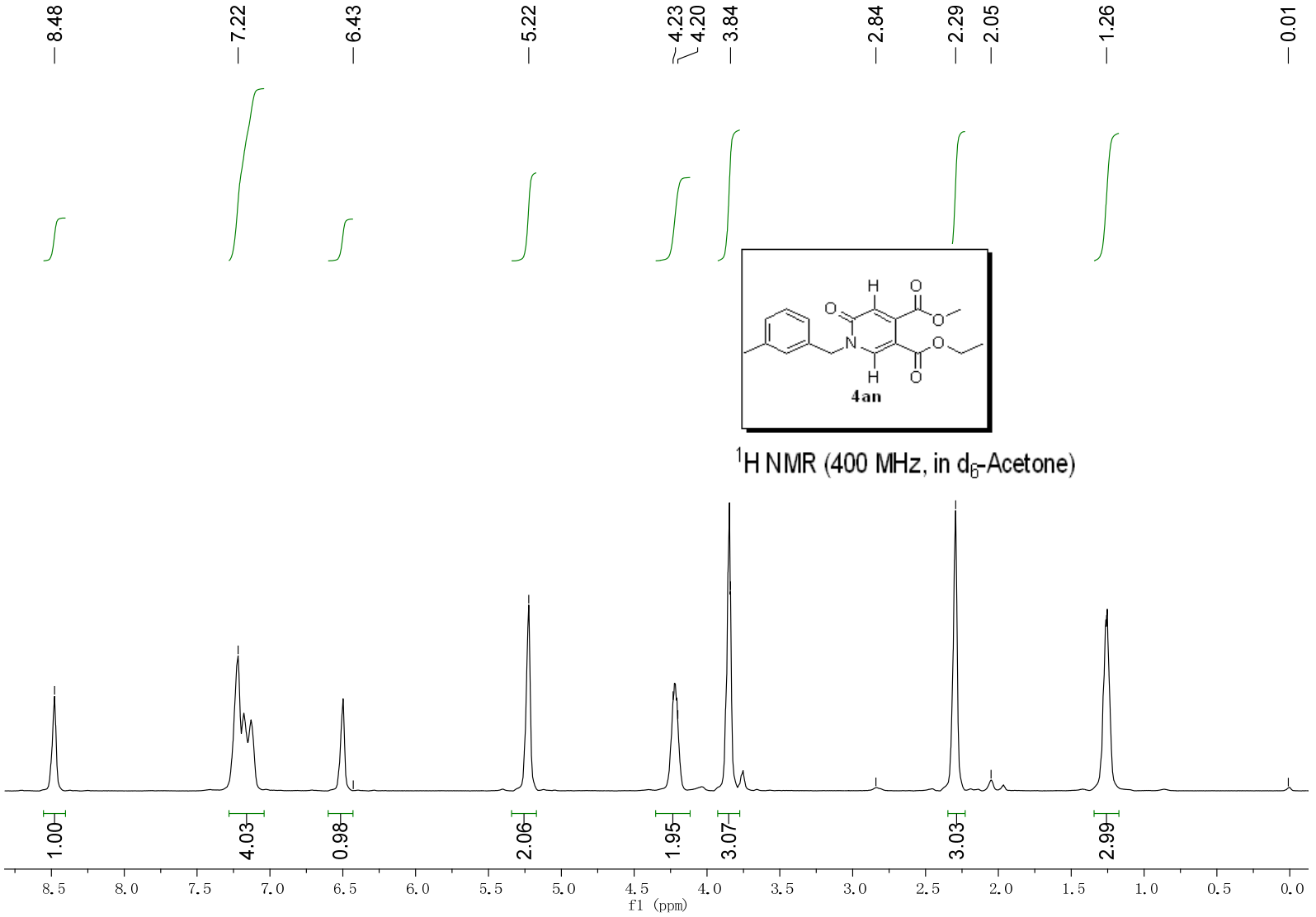


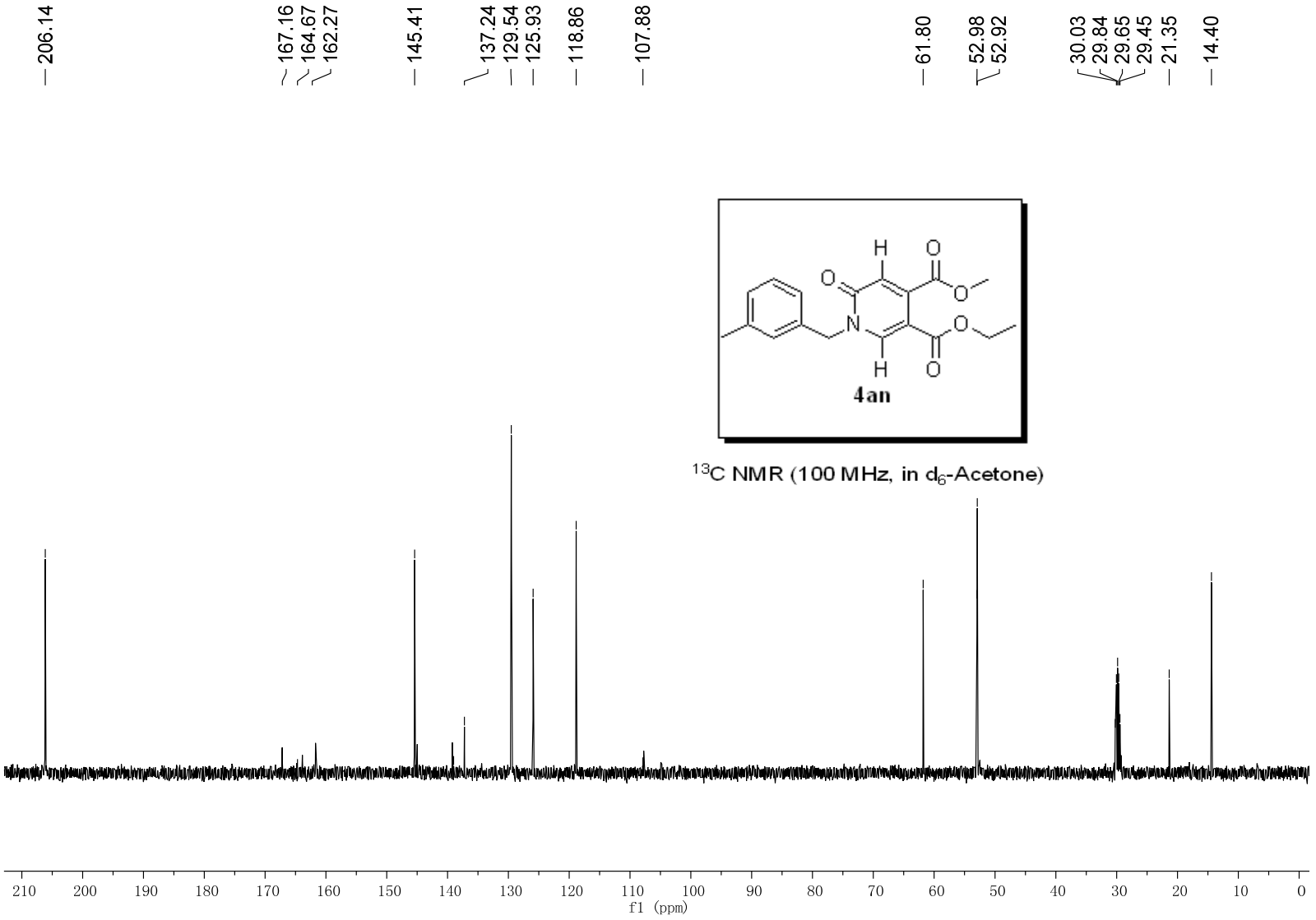


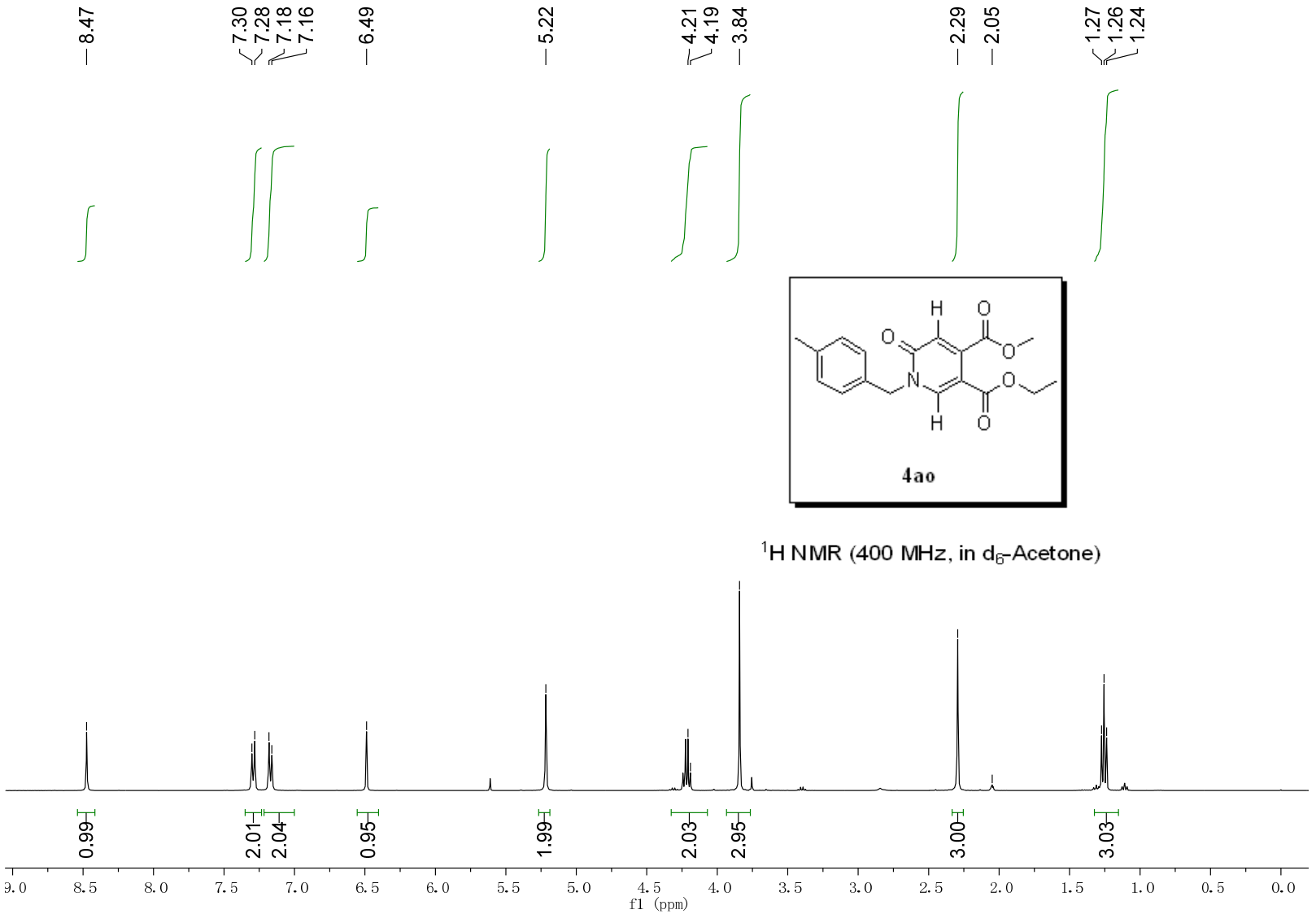




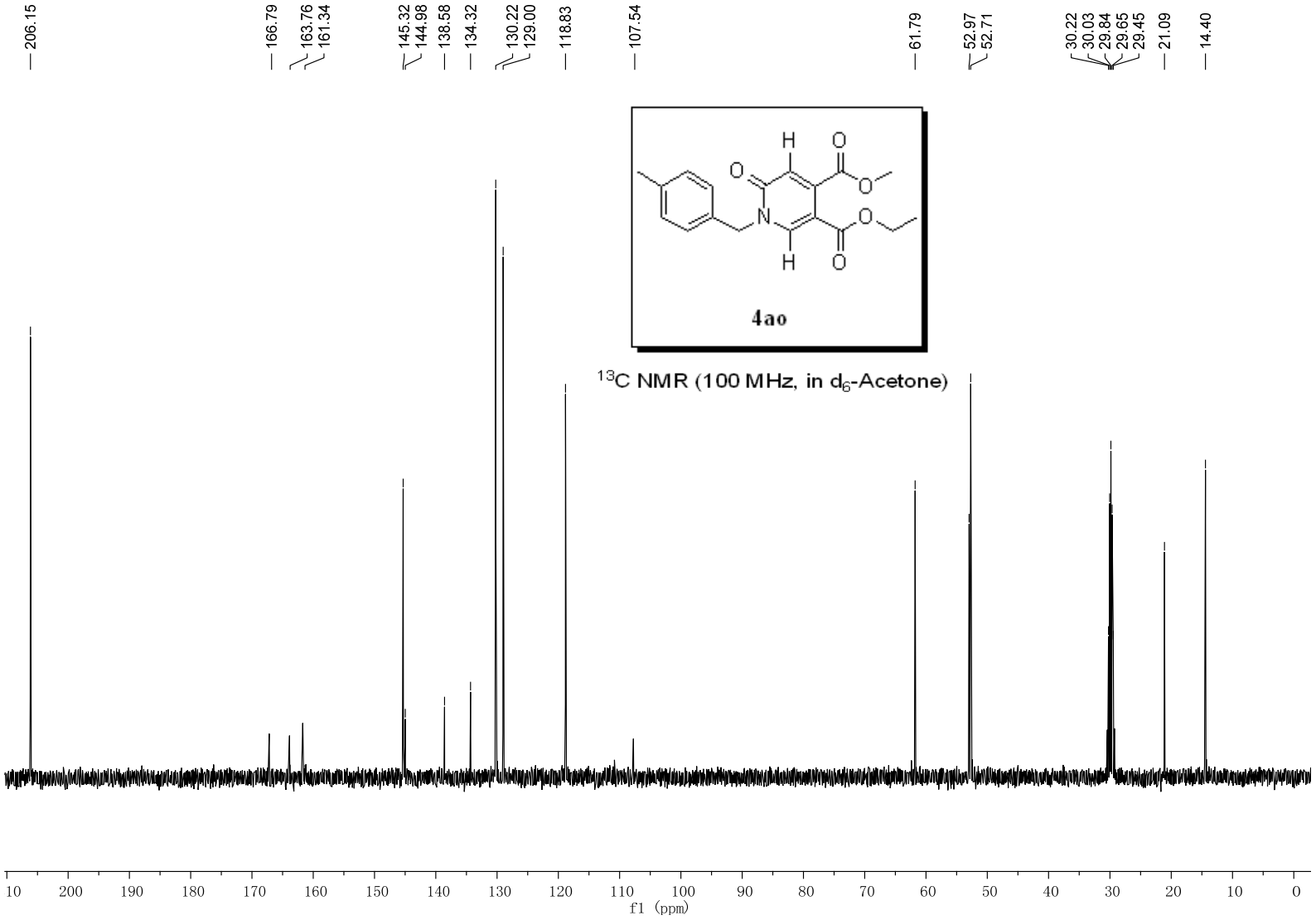


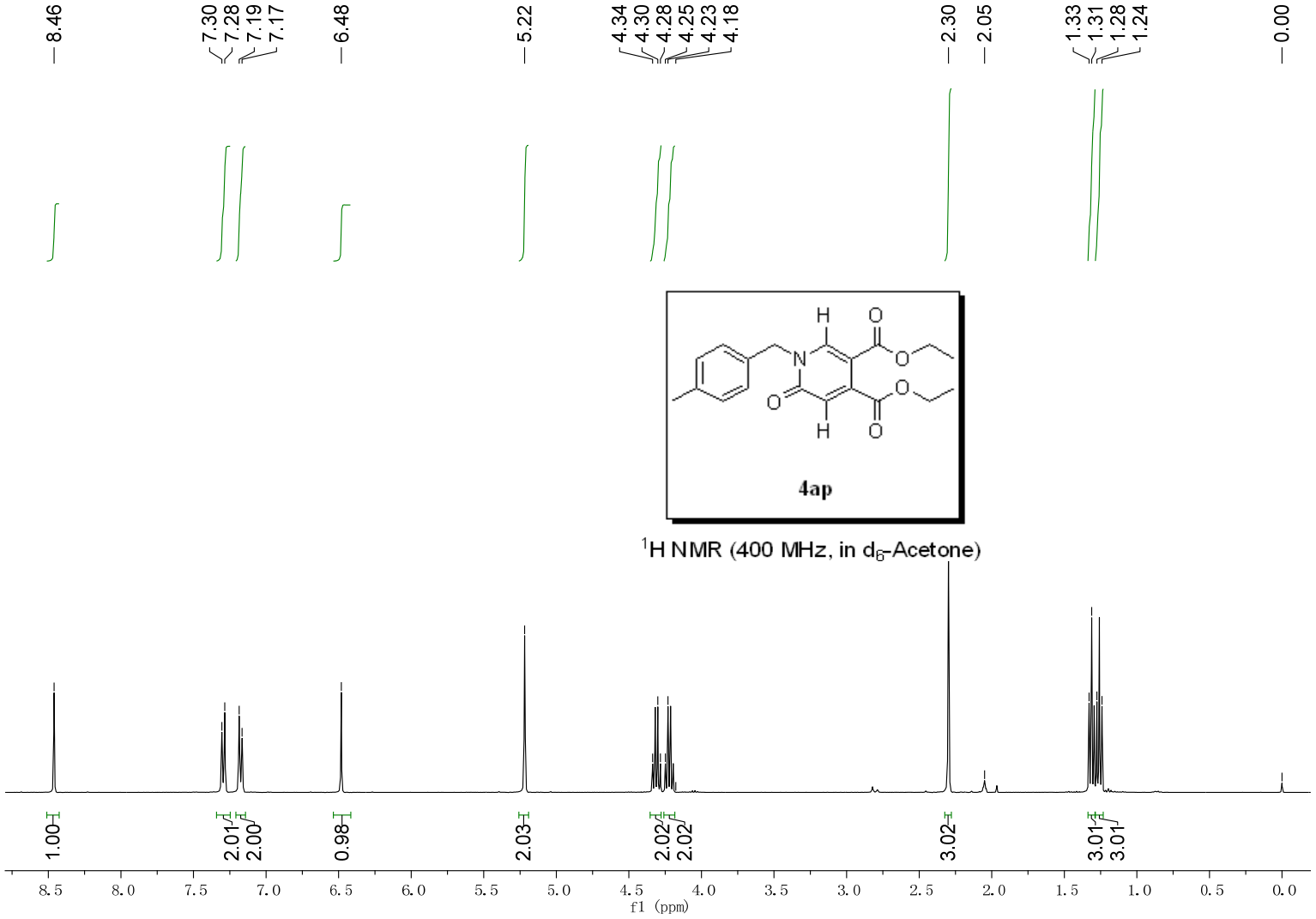


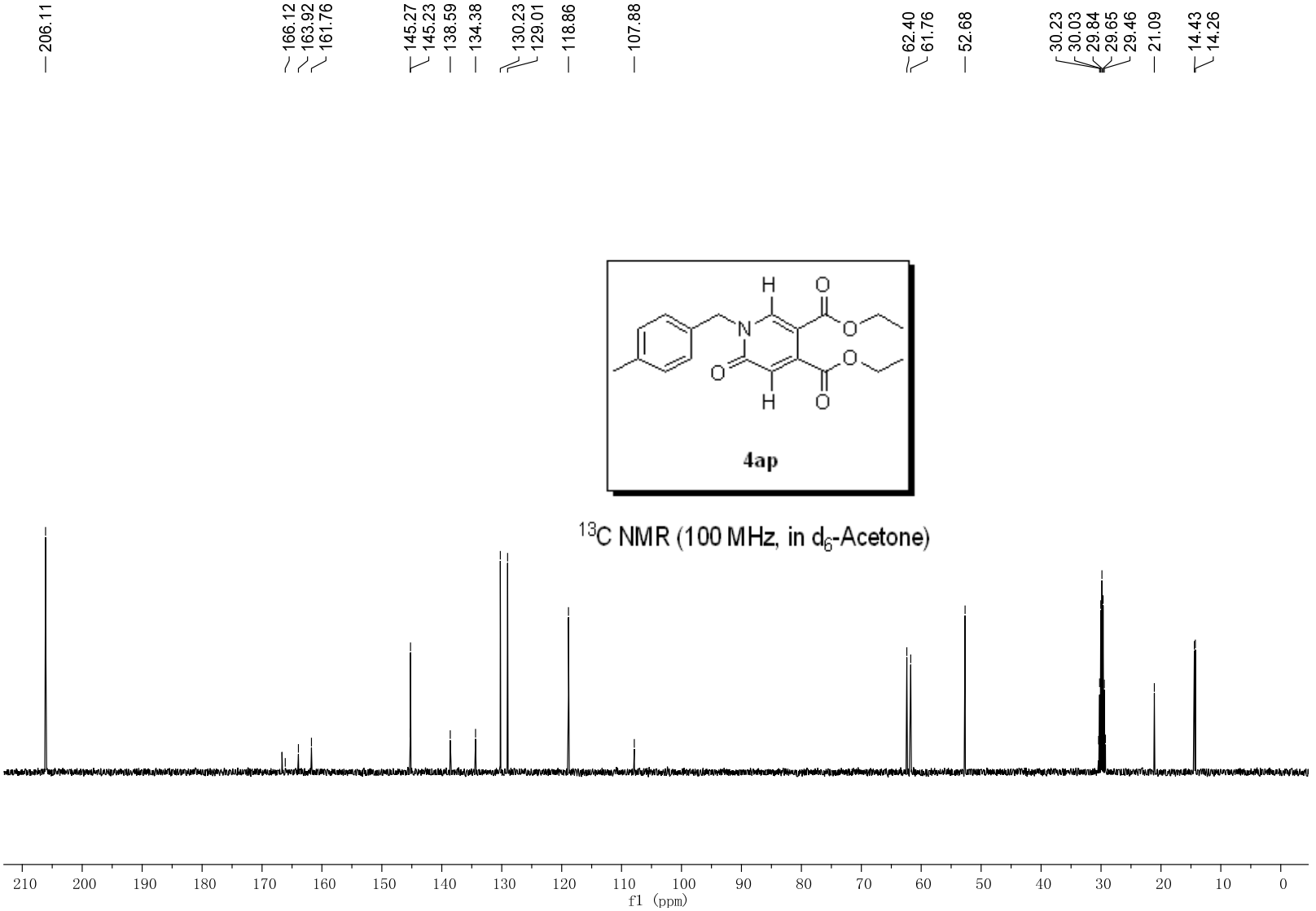


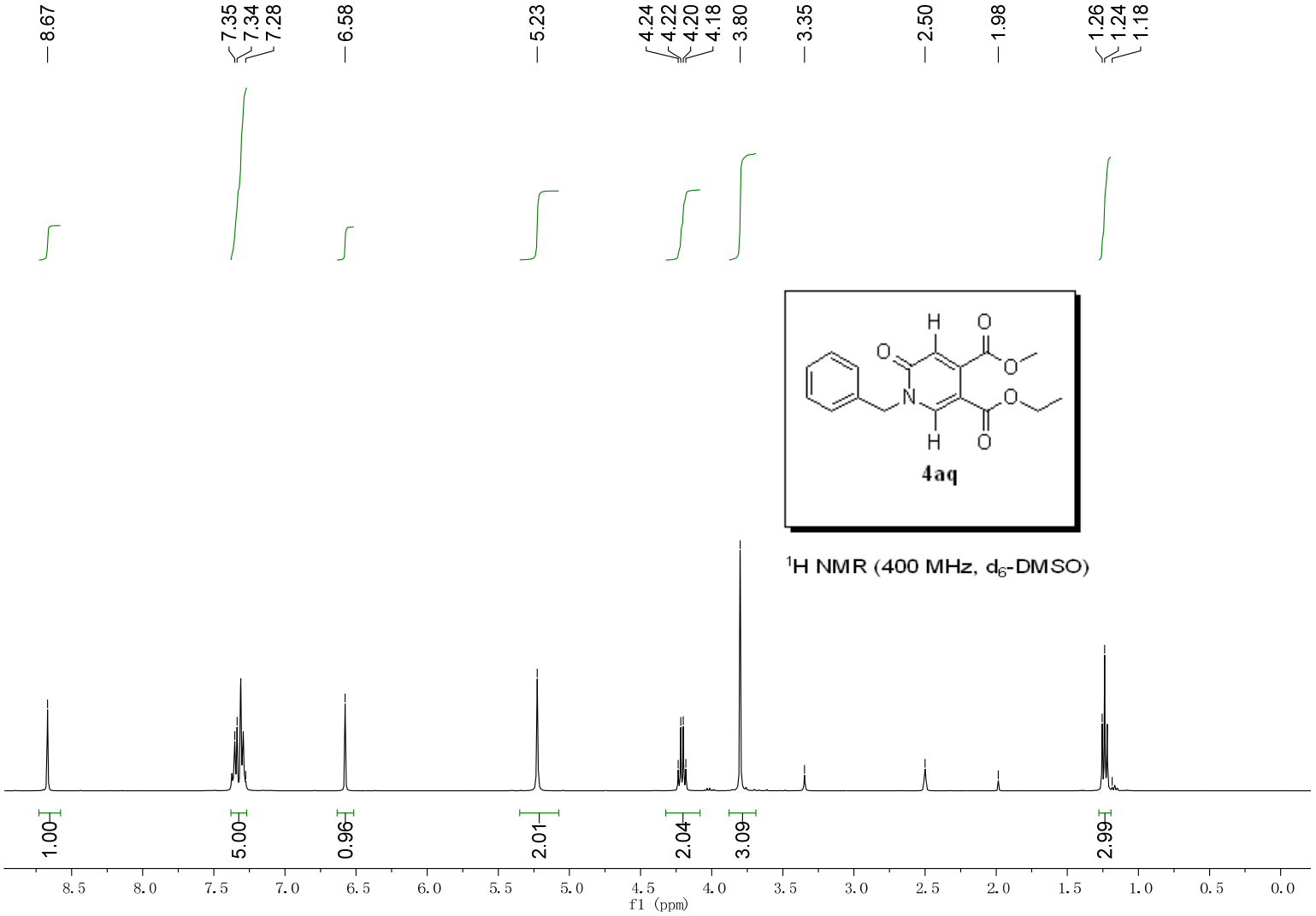


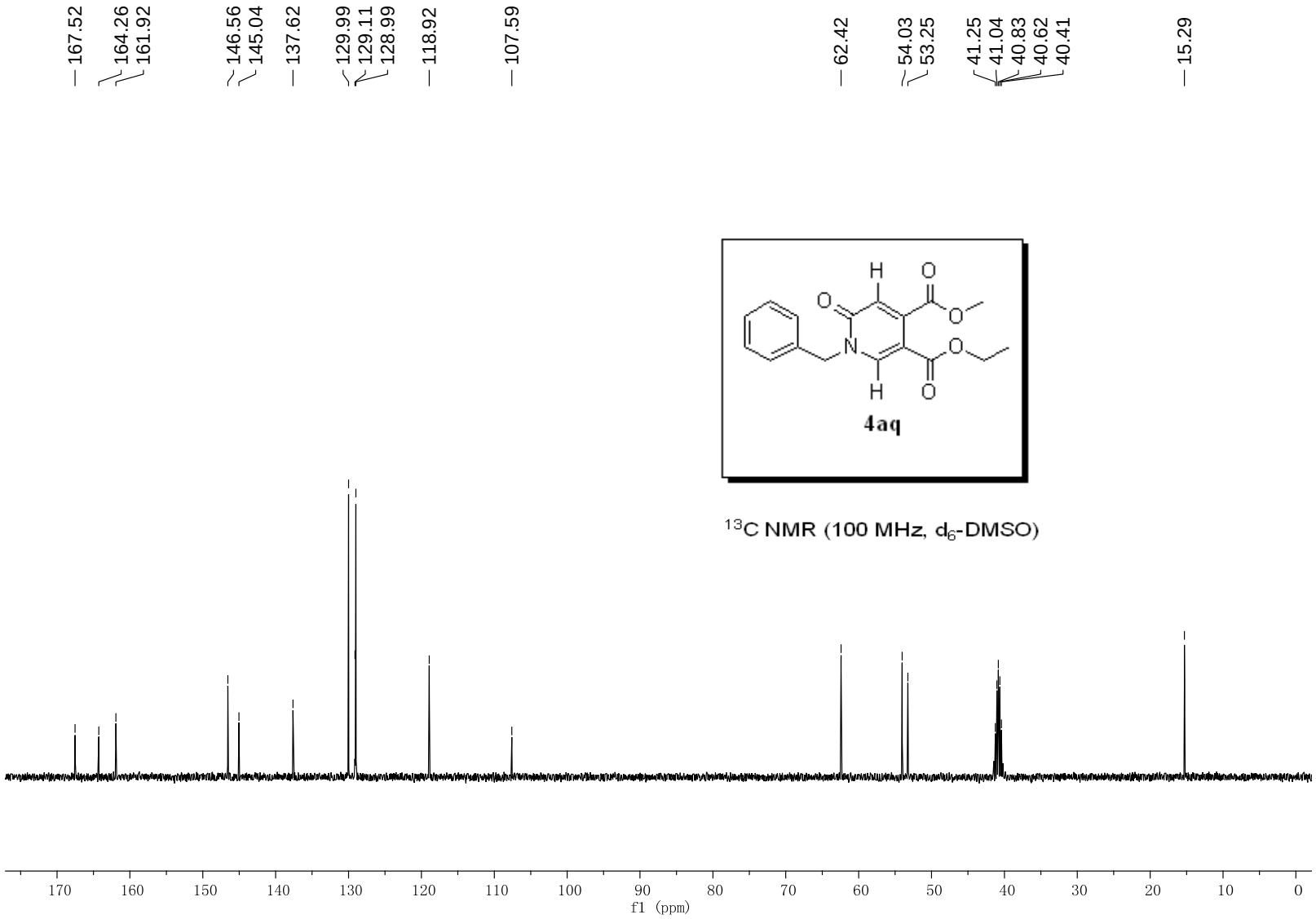
^{1}H NMR (400 MHz, in d_6-Acetone)

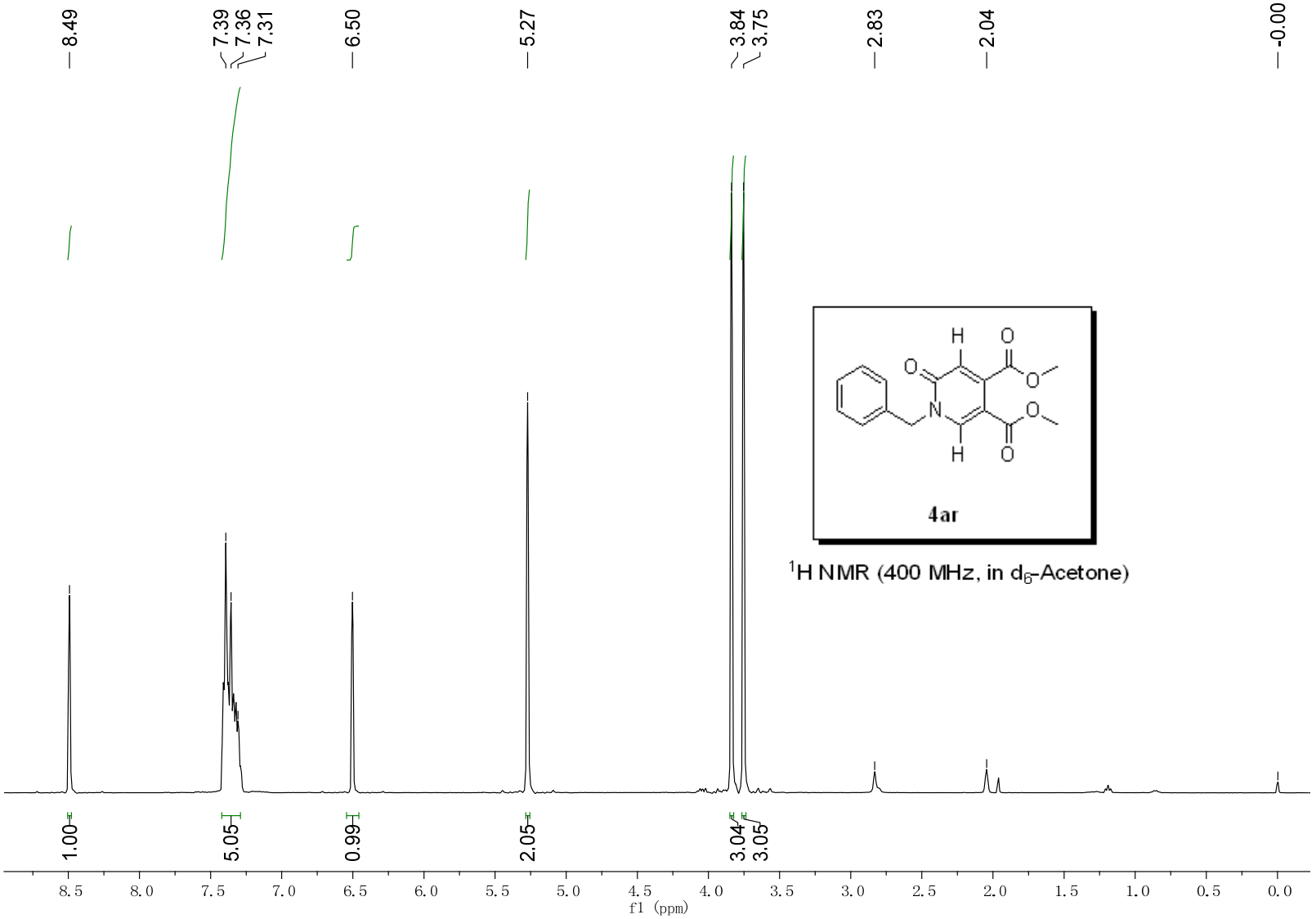


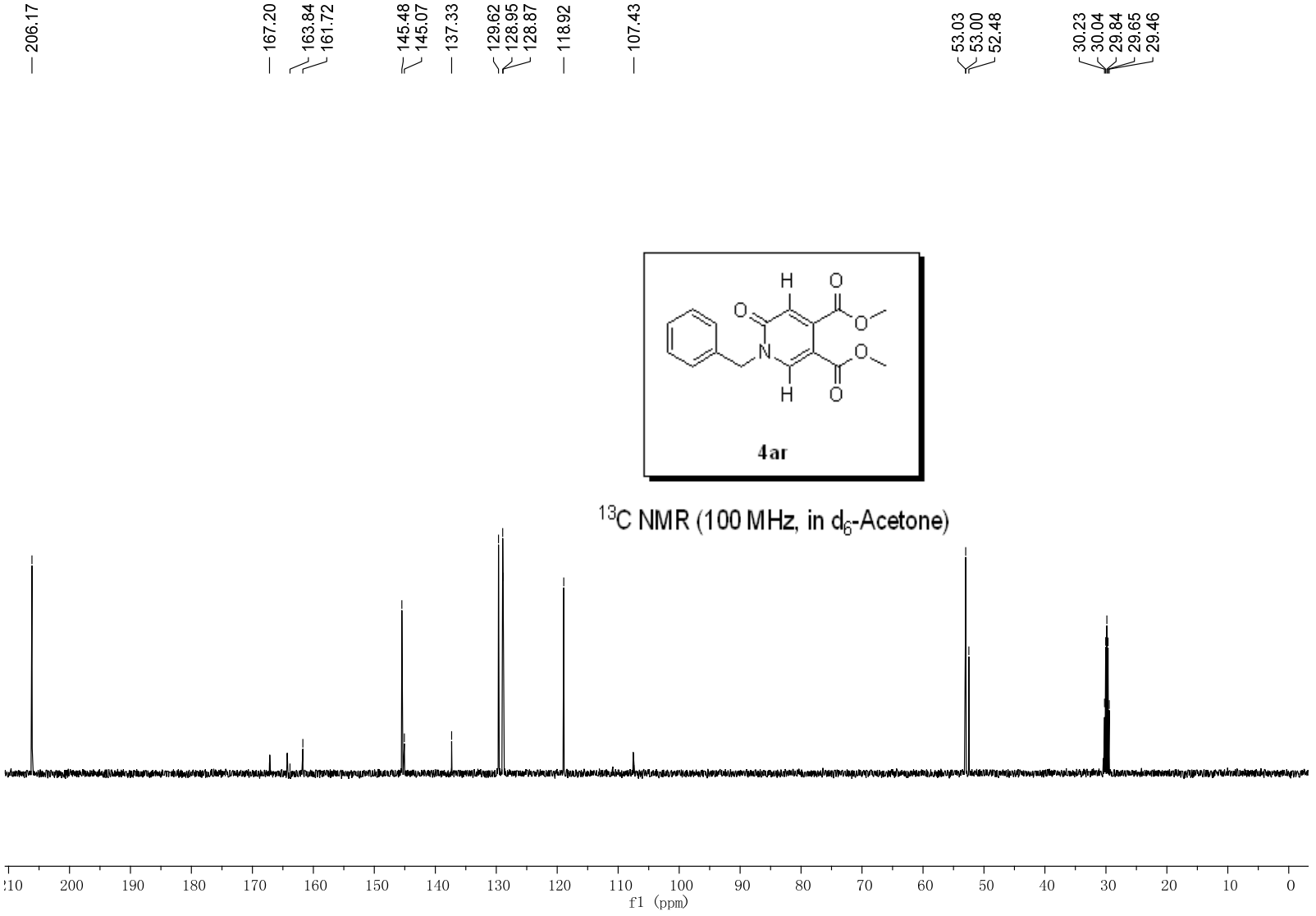


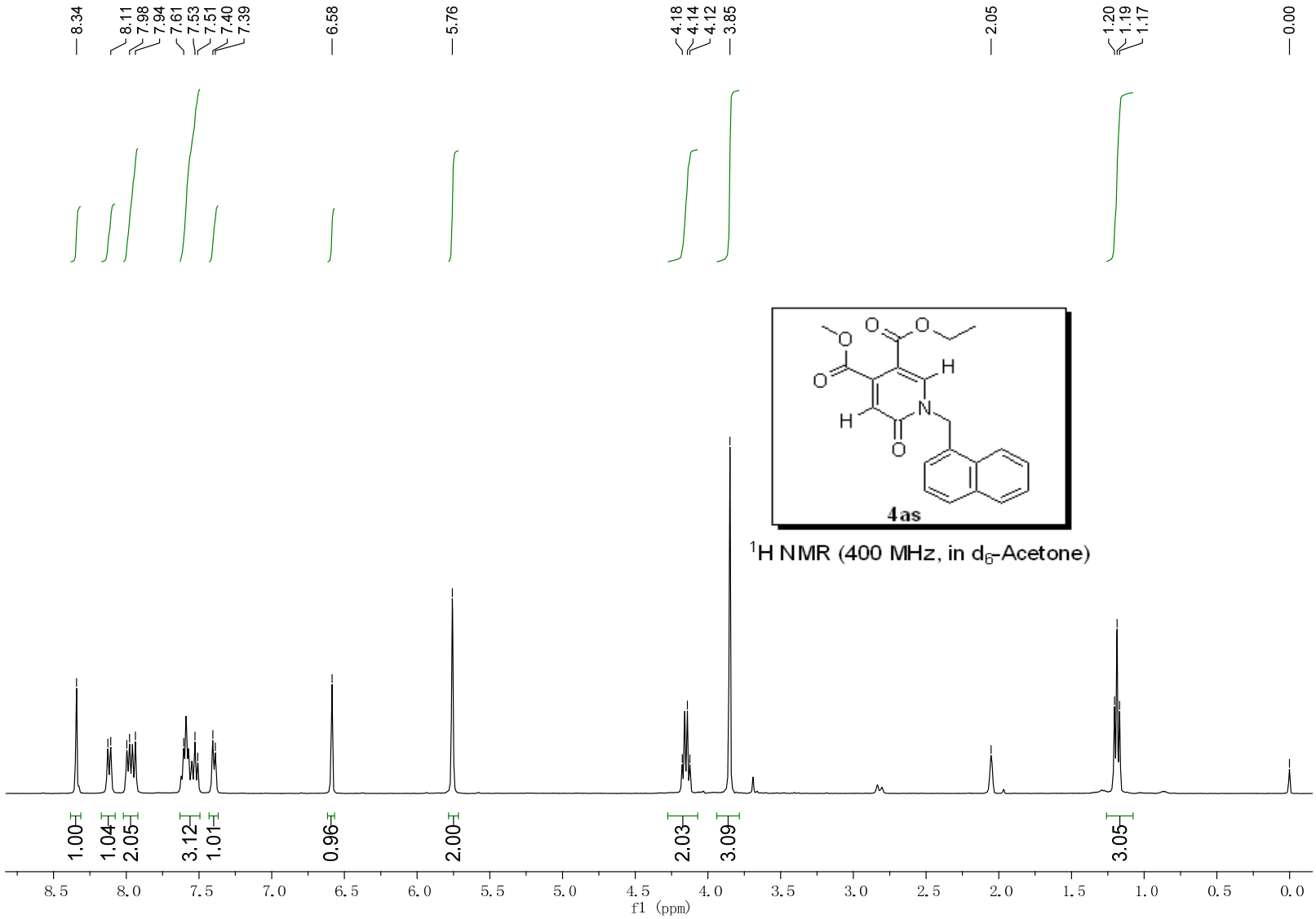


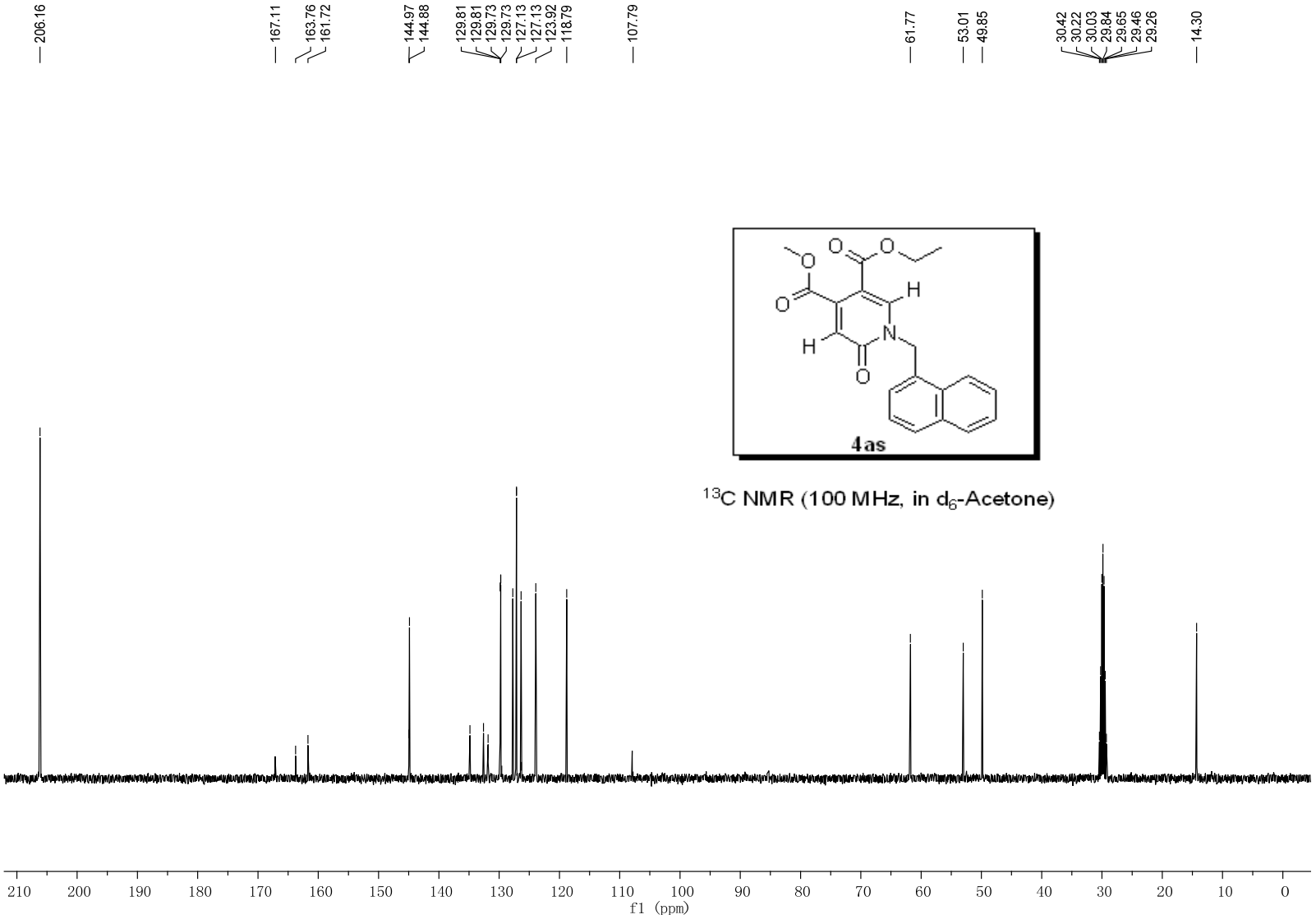


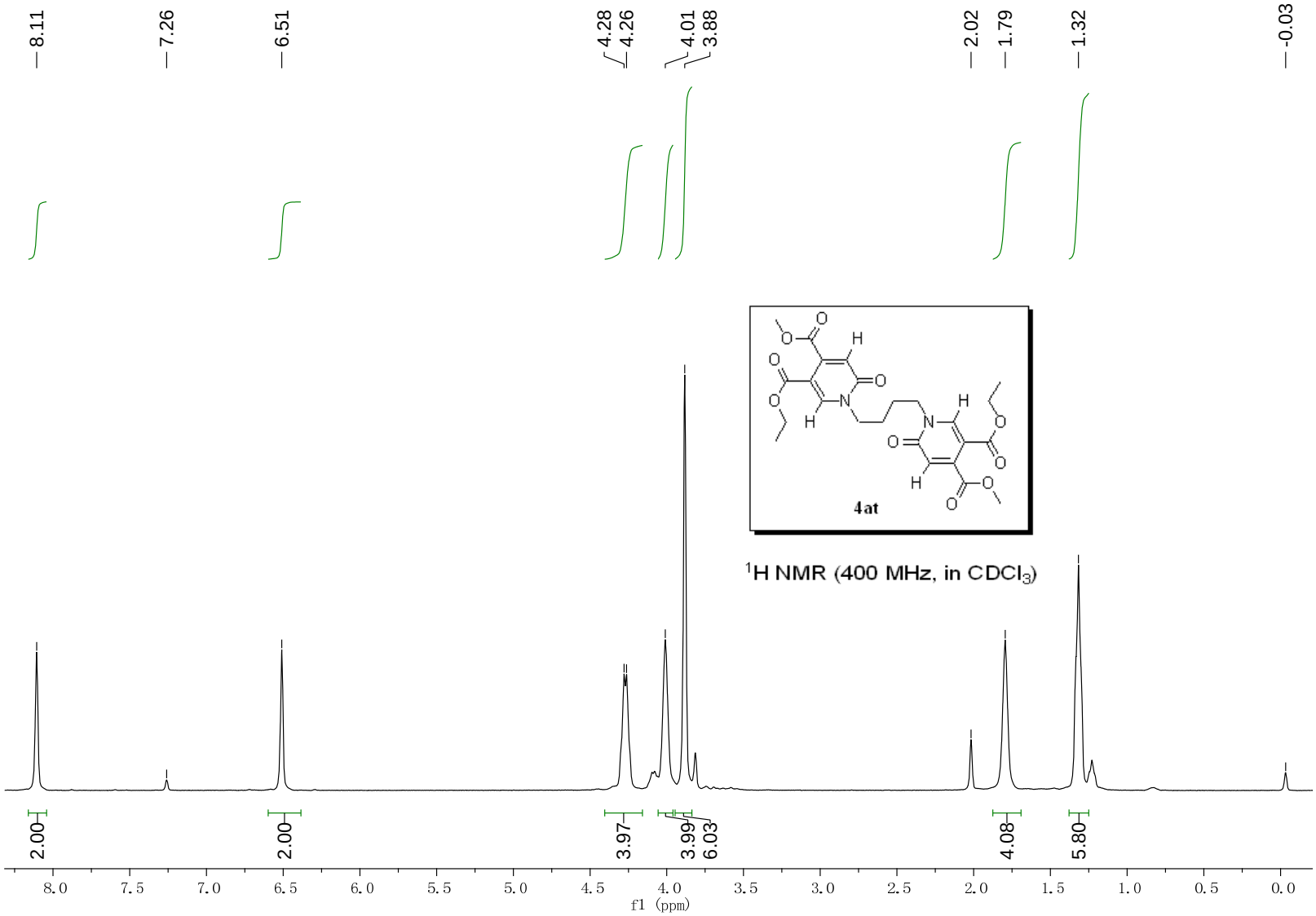


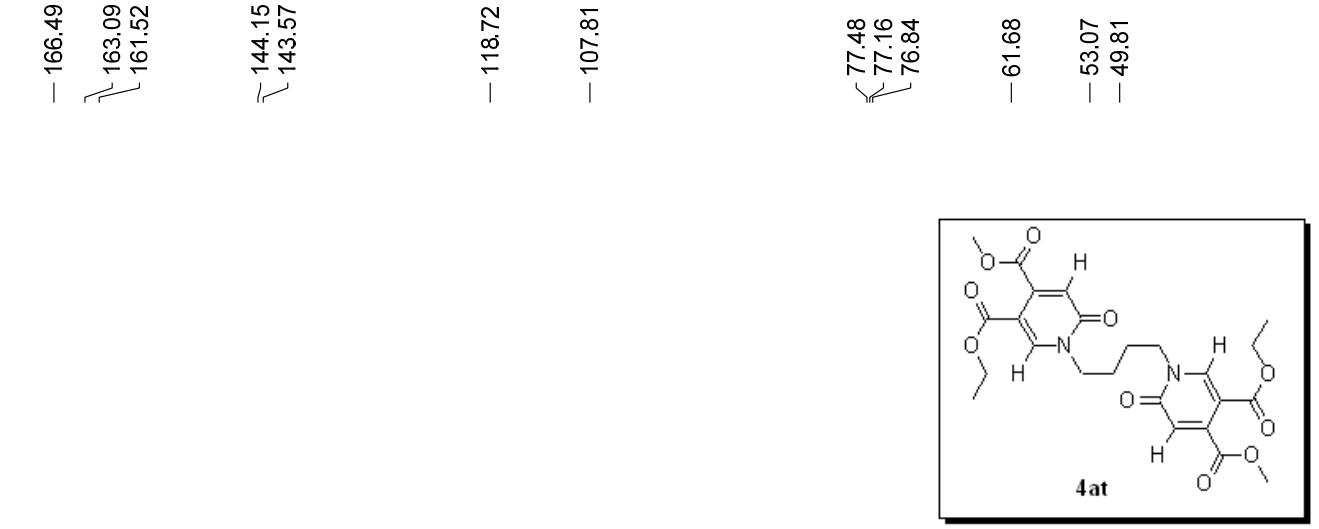




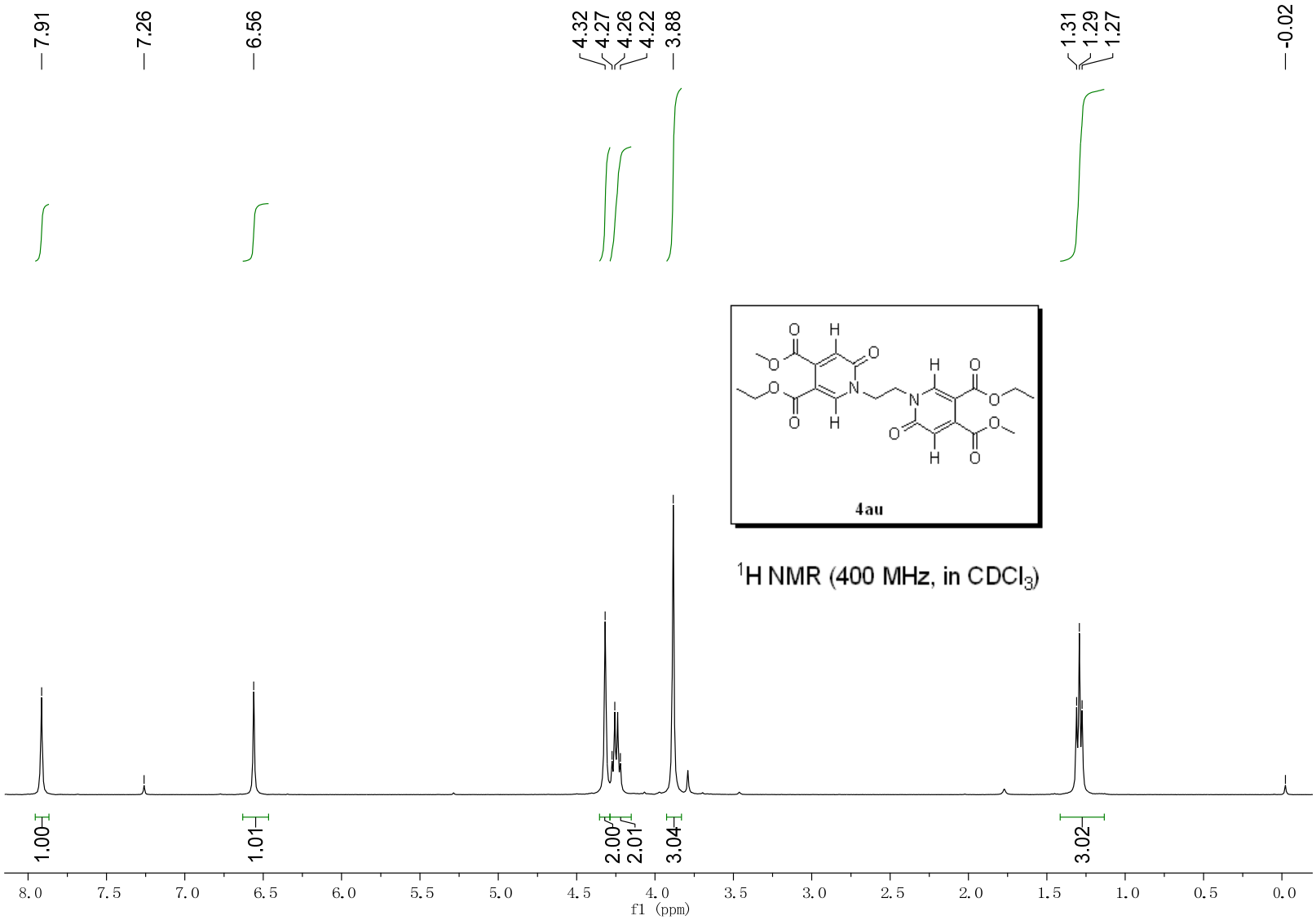


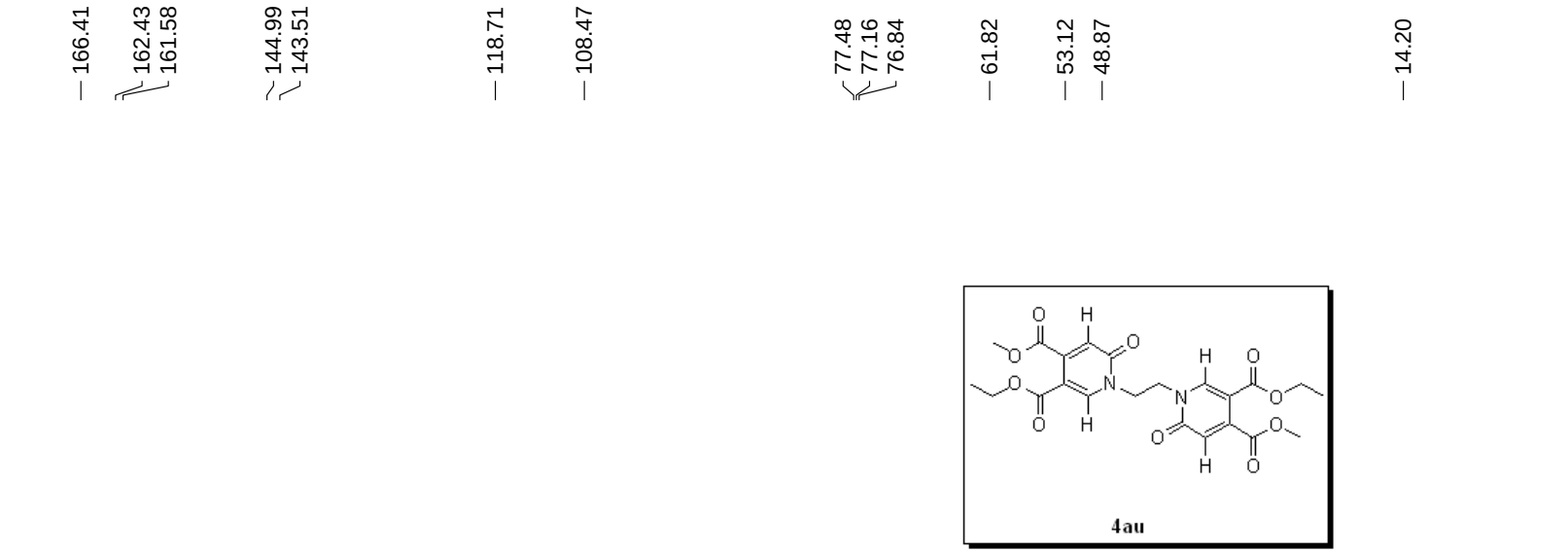


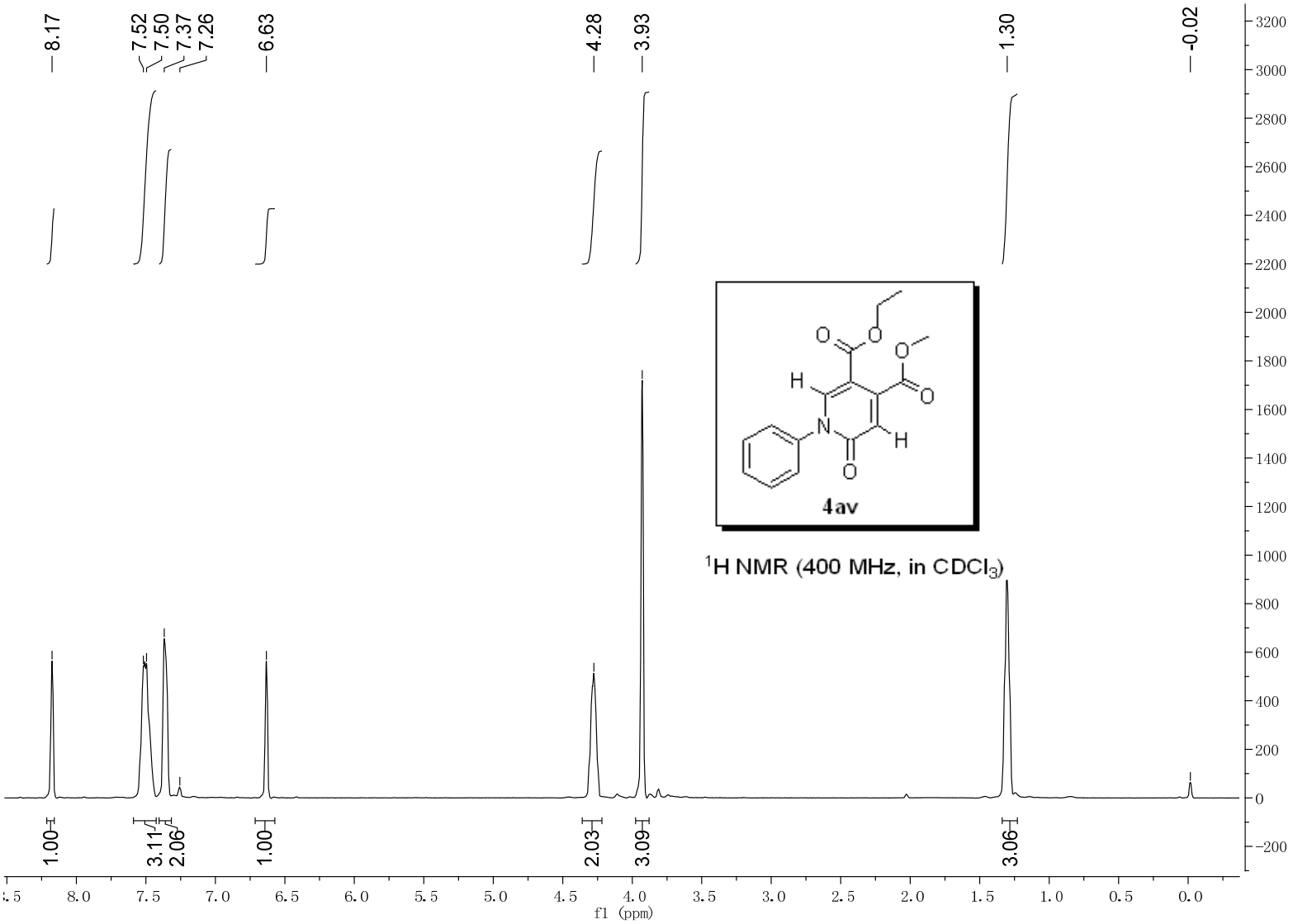


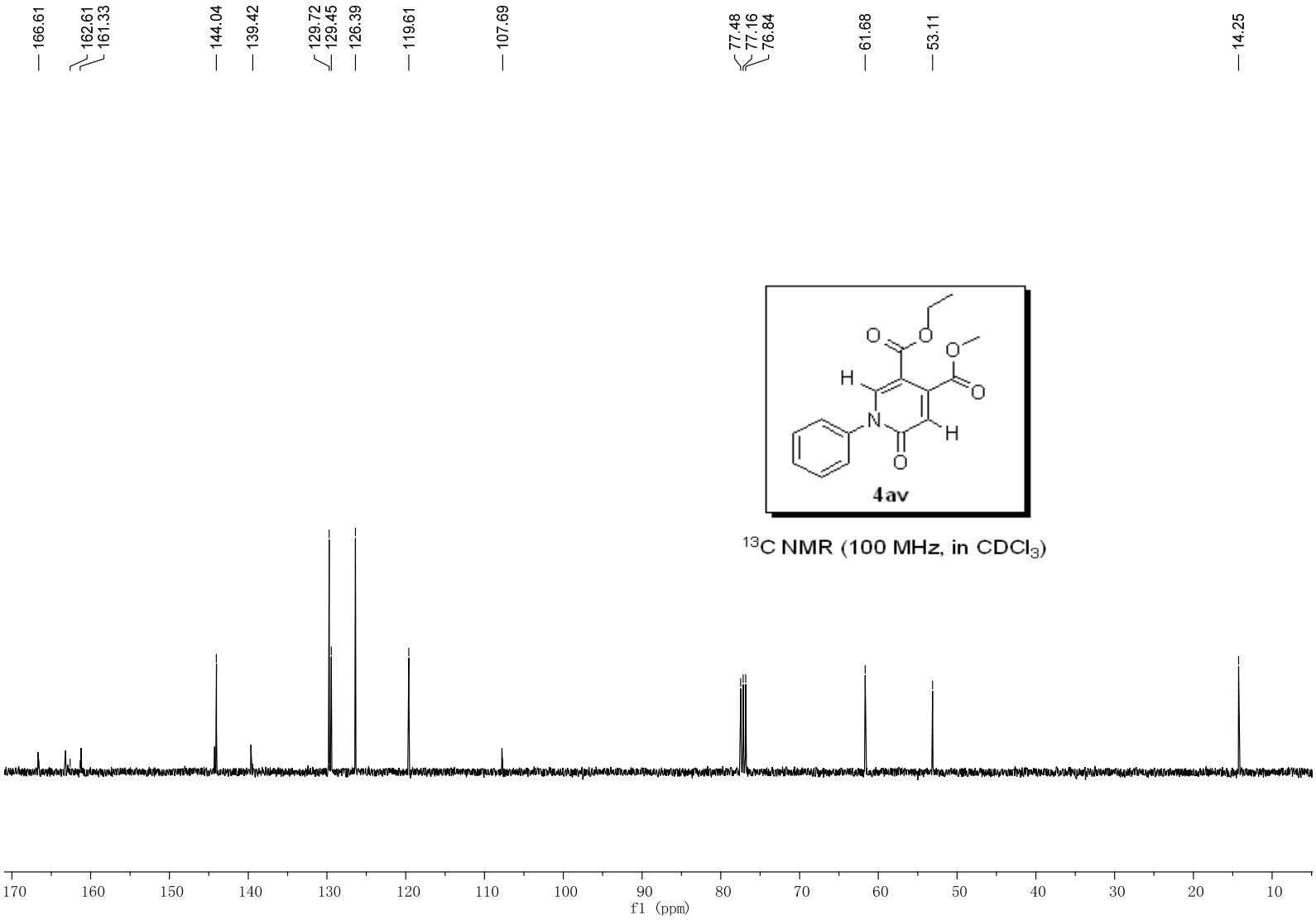


^{13}C NMR (100 MHz, in $CDCl_3$)








166.61
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129.72
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107.69
77.48
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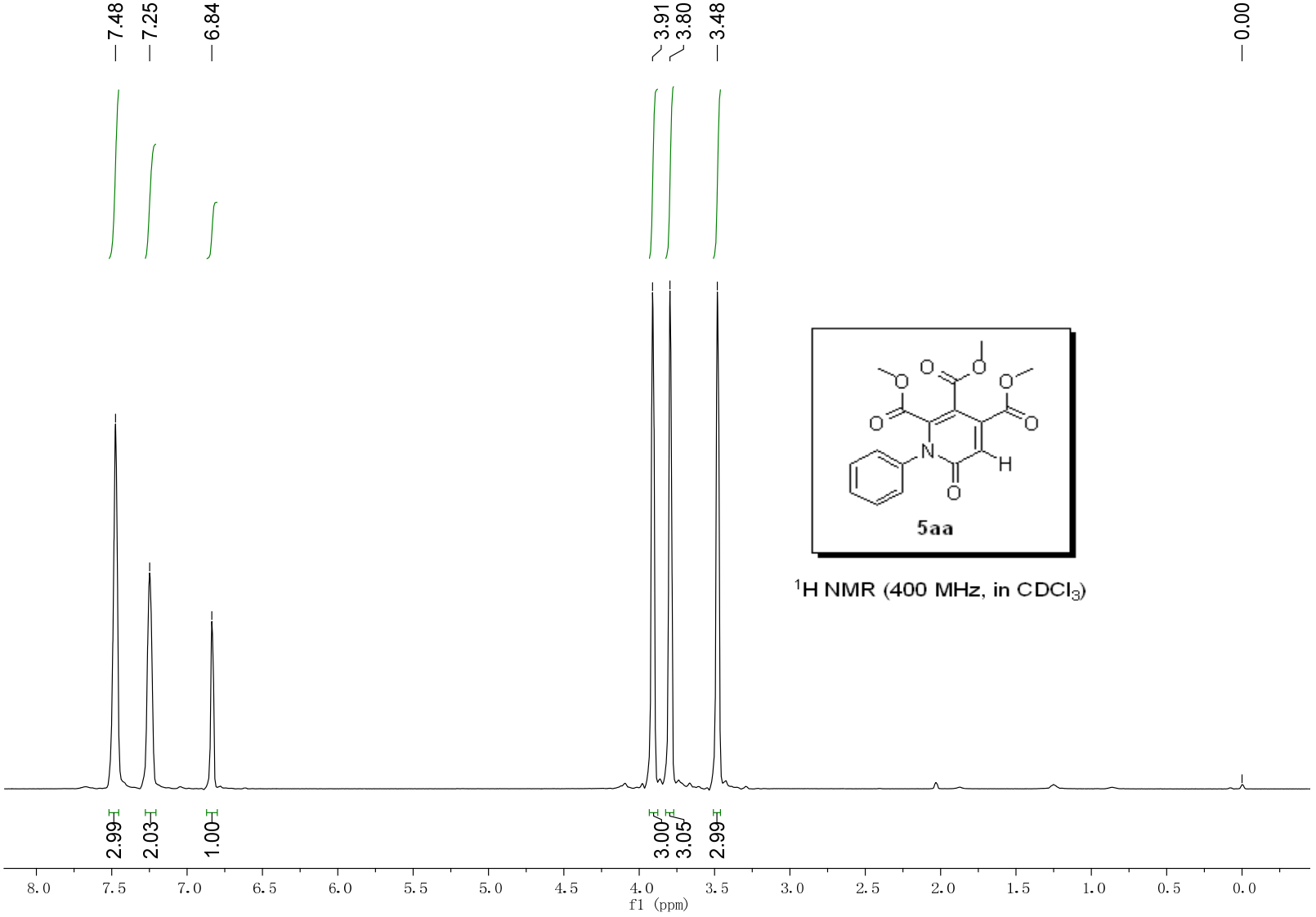
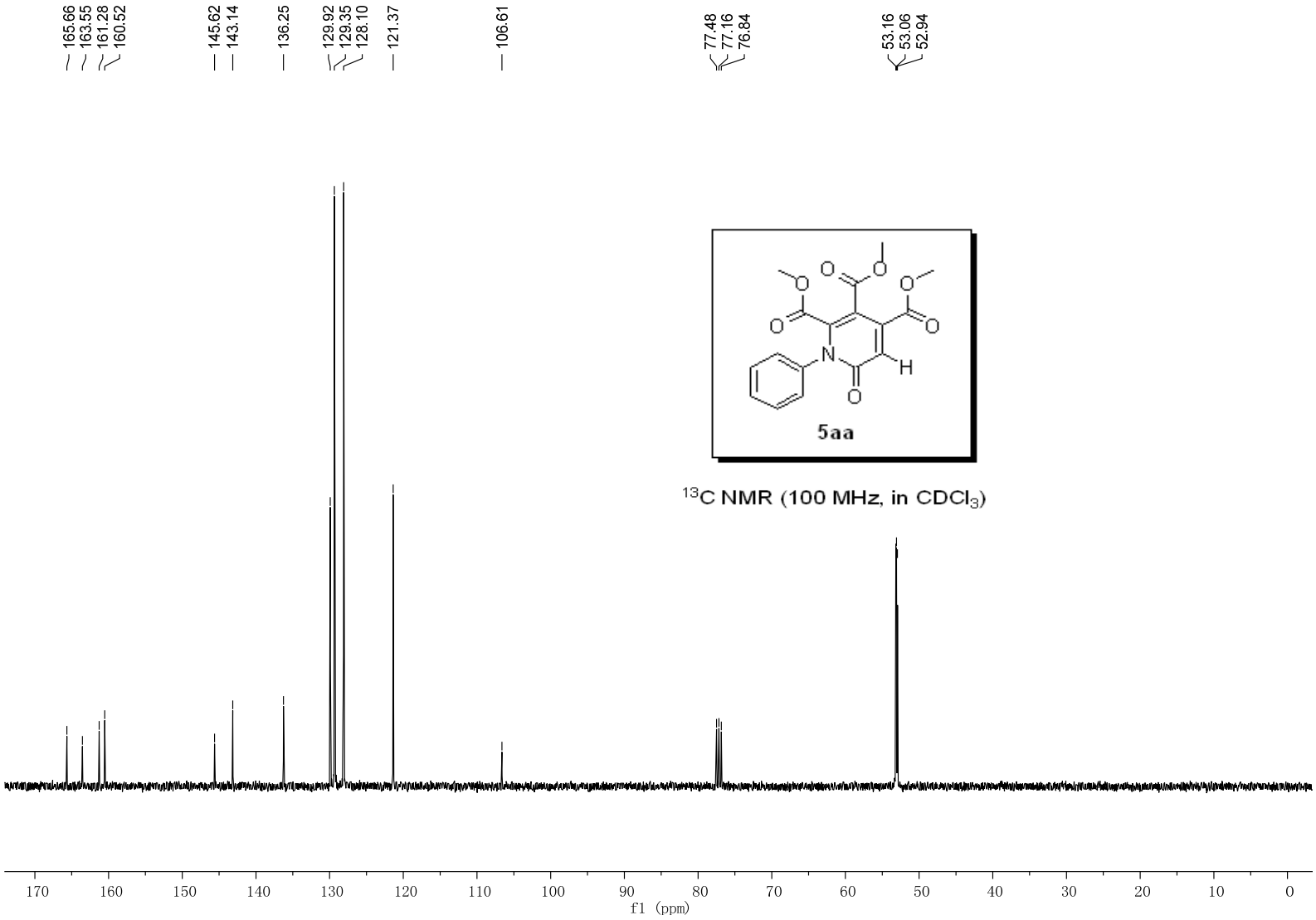
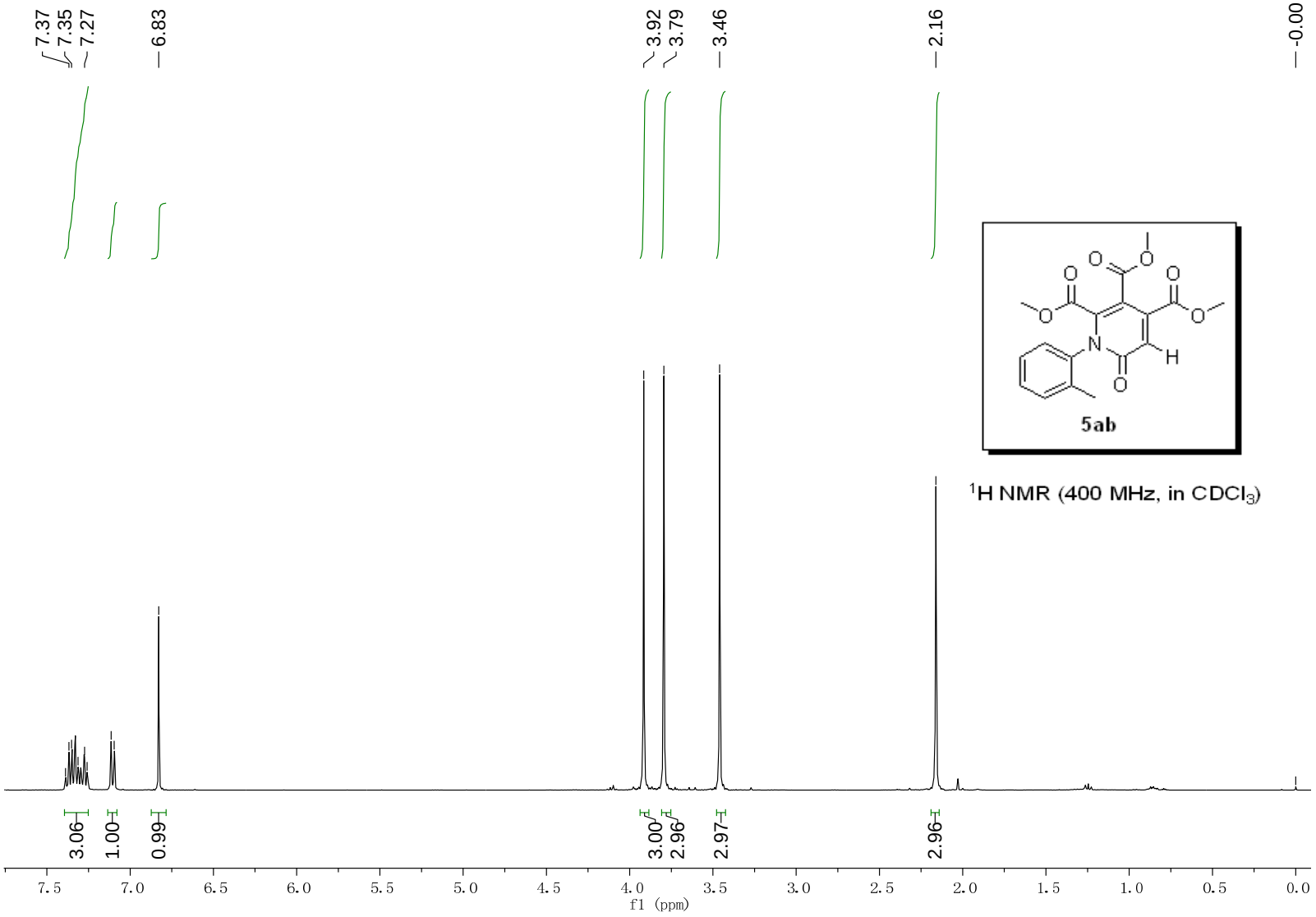
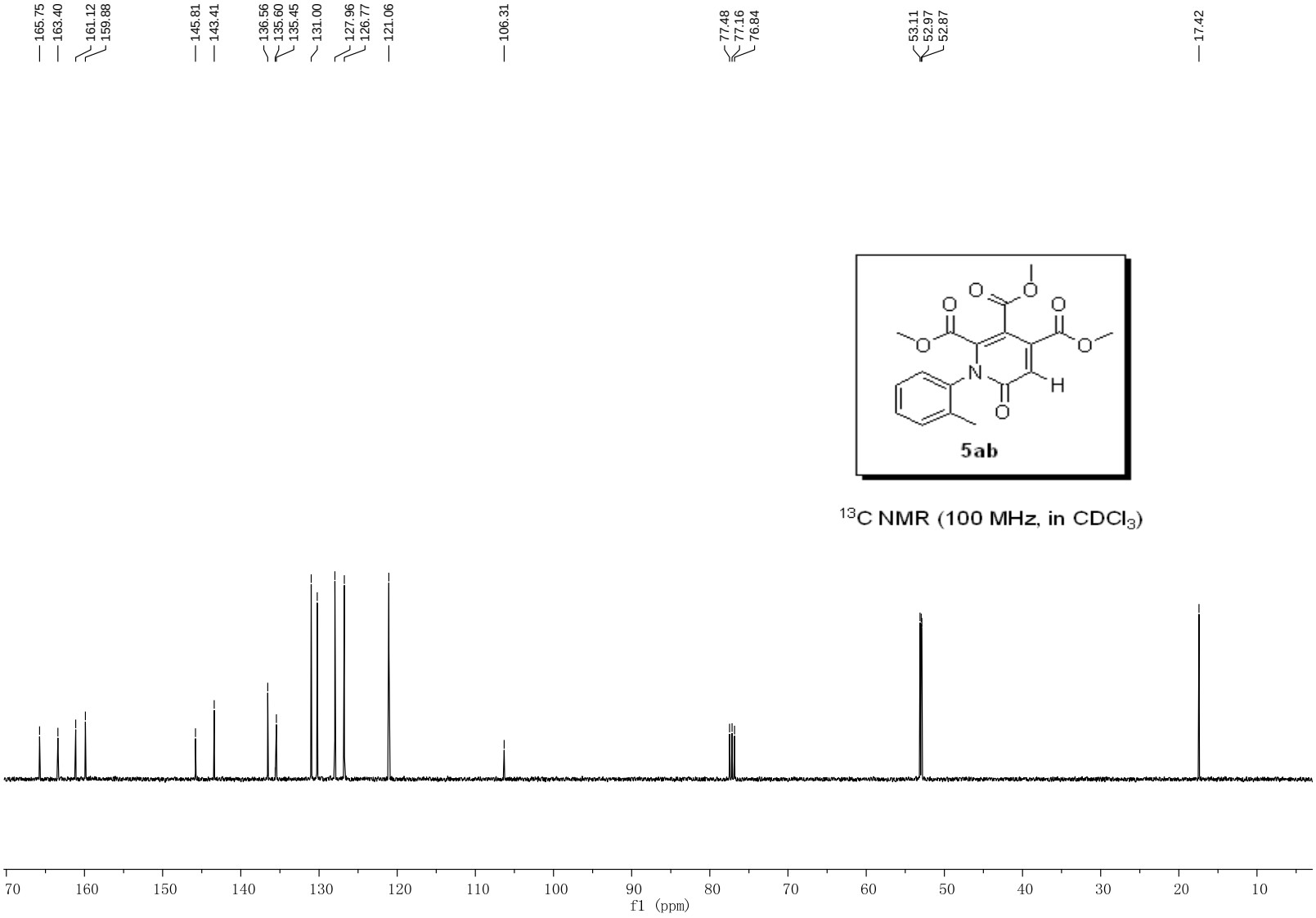
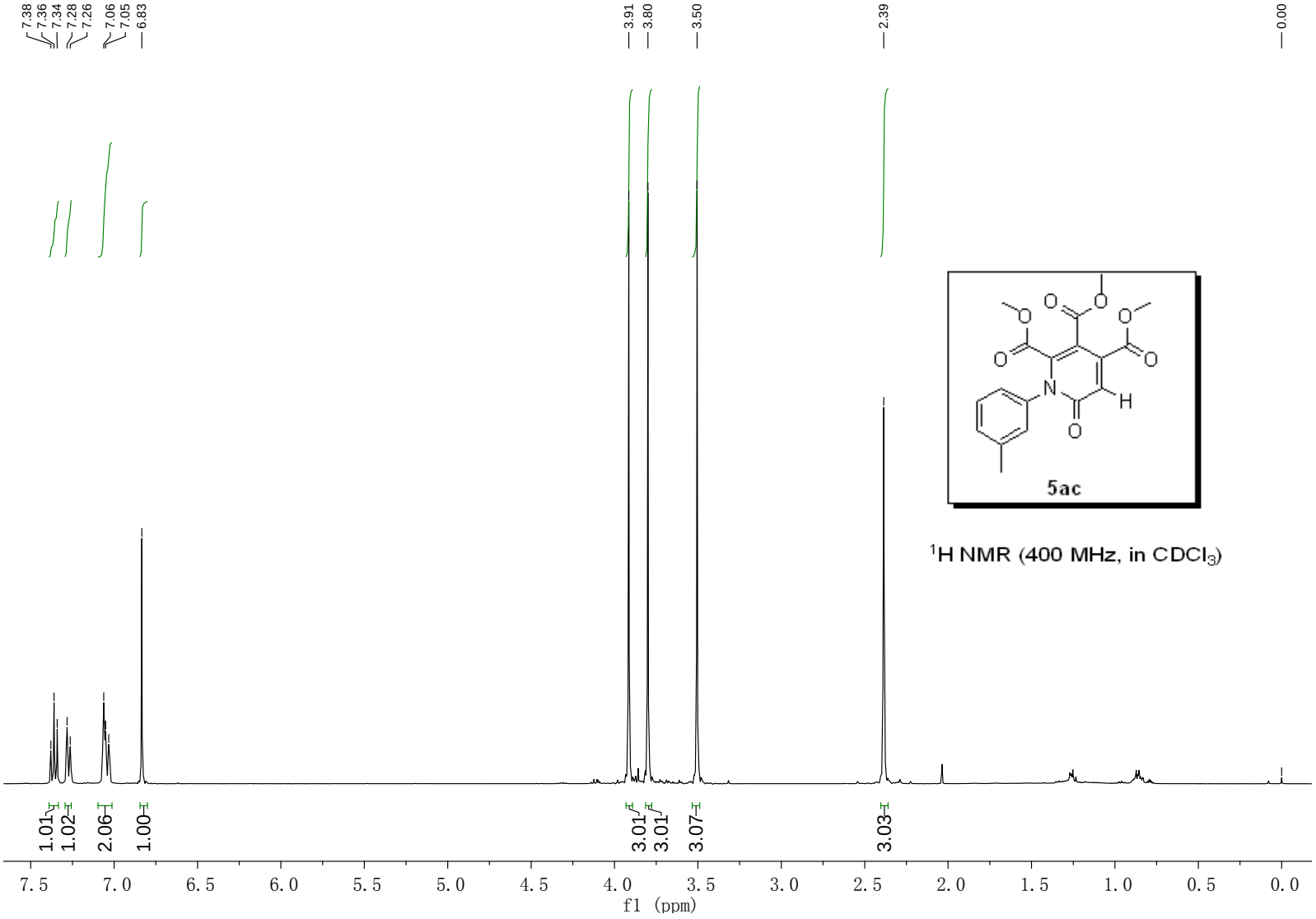
76.84
61.68
53.11
14.25
4av
13C NMR (100 MHz, in CDCl3)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

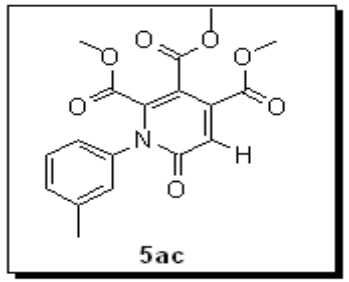






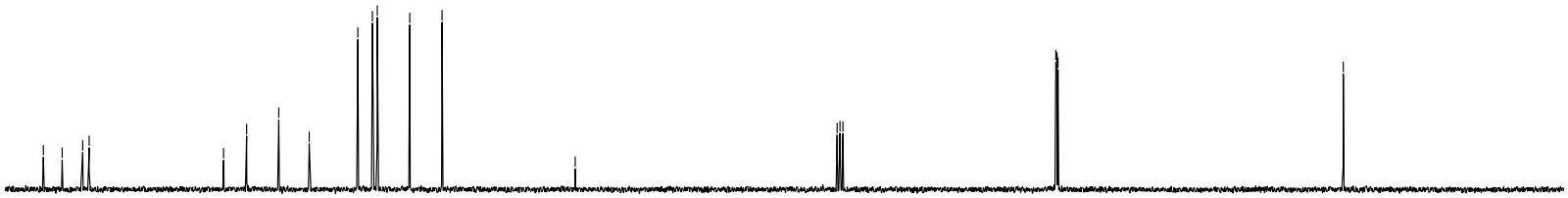


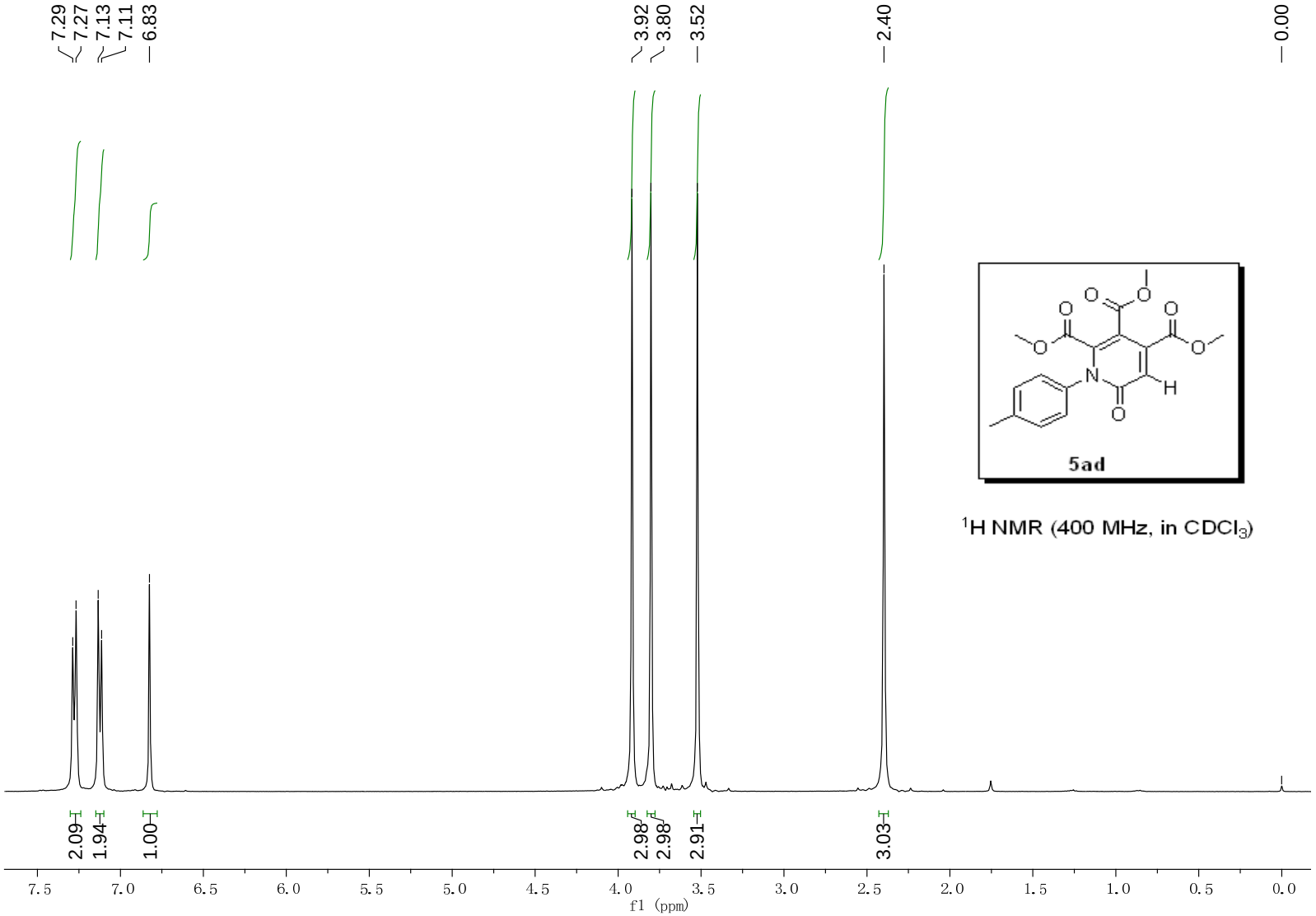


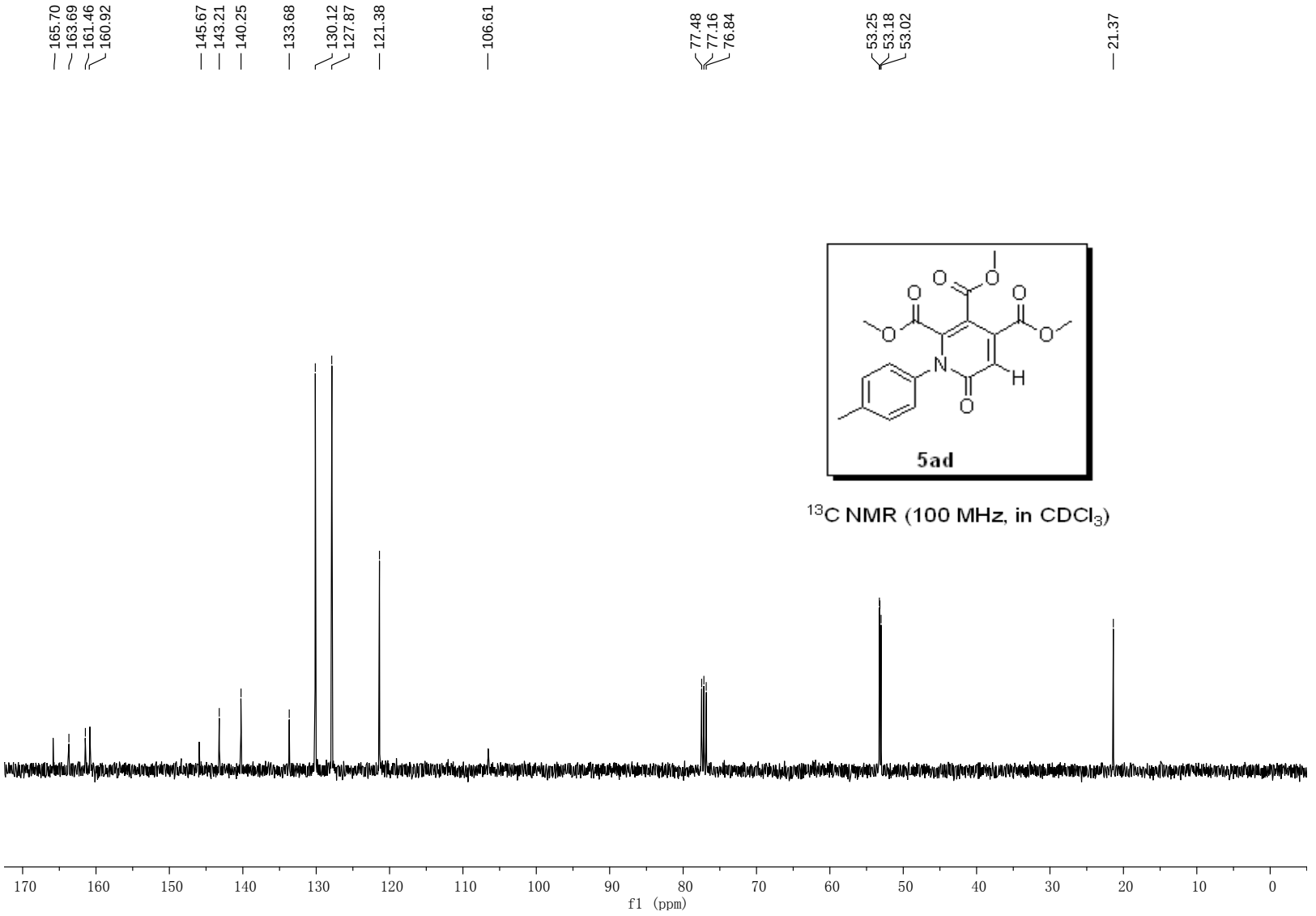


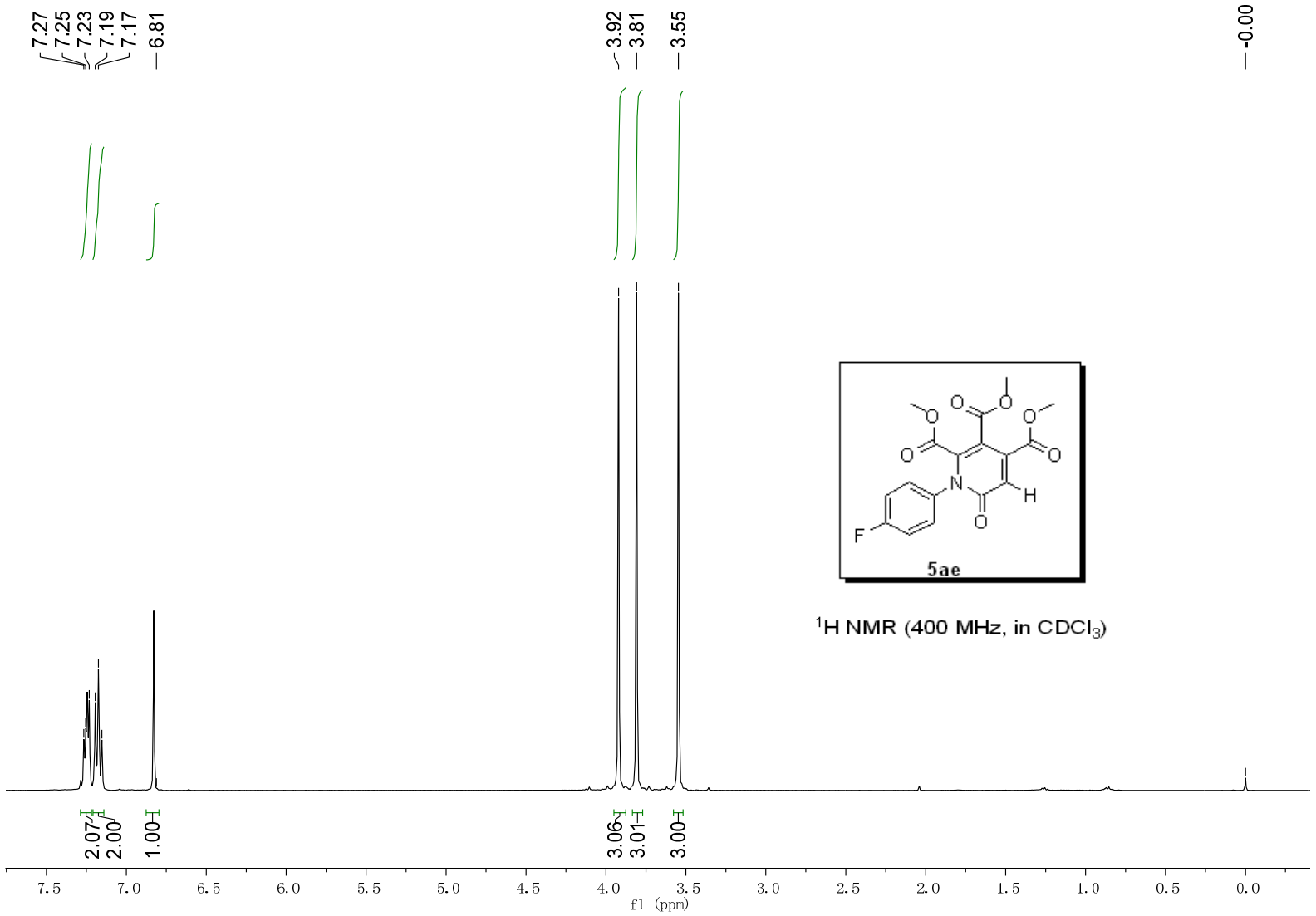
5ac

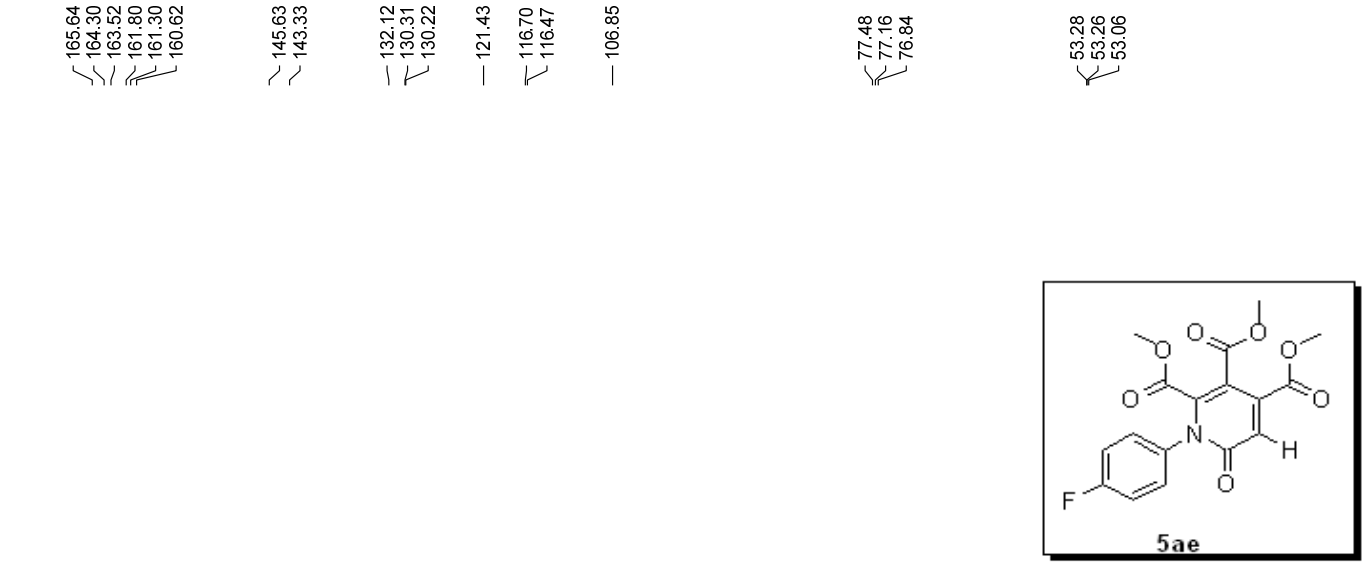
^{13}C NMR (100 MHz, in $CDCl_3$)

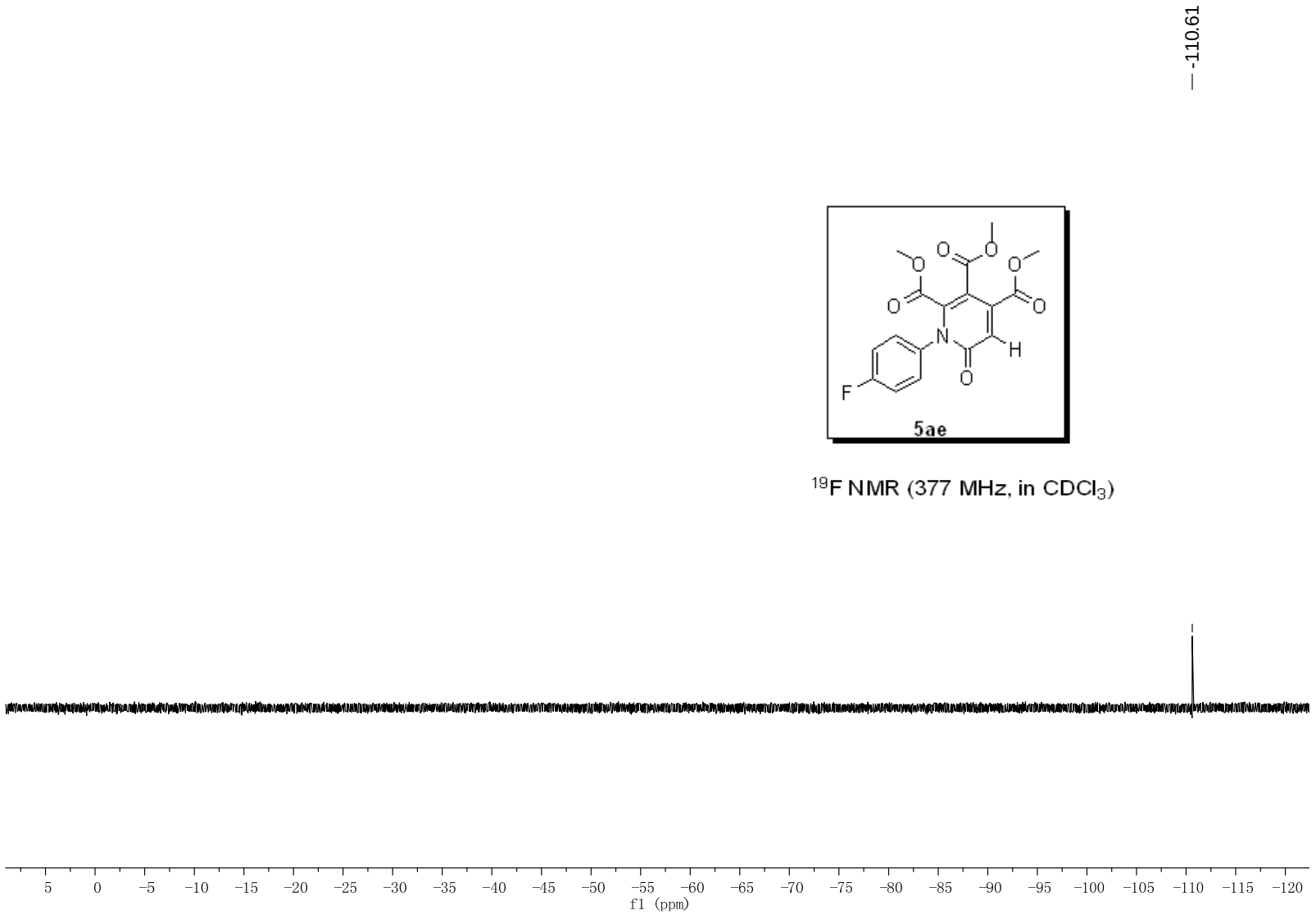


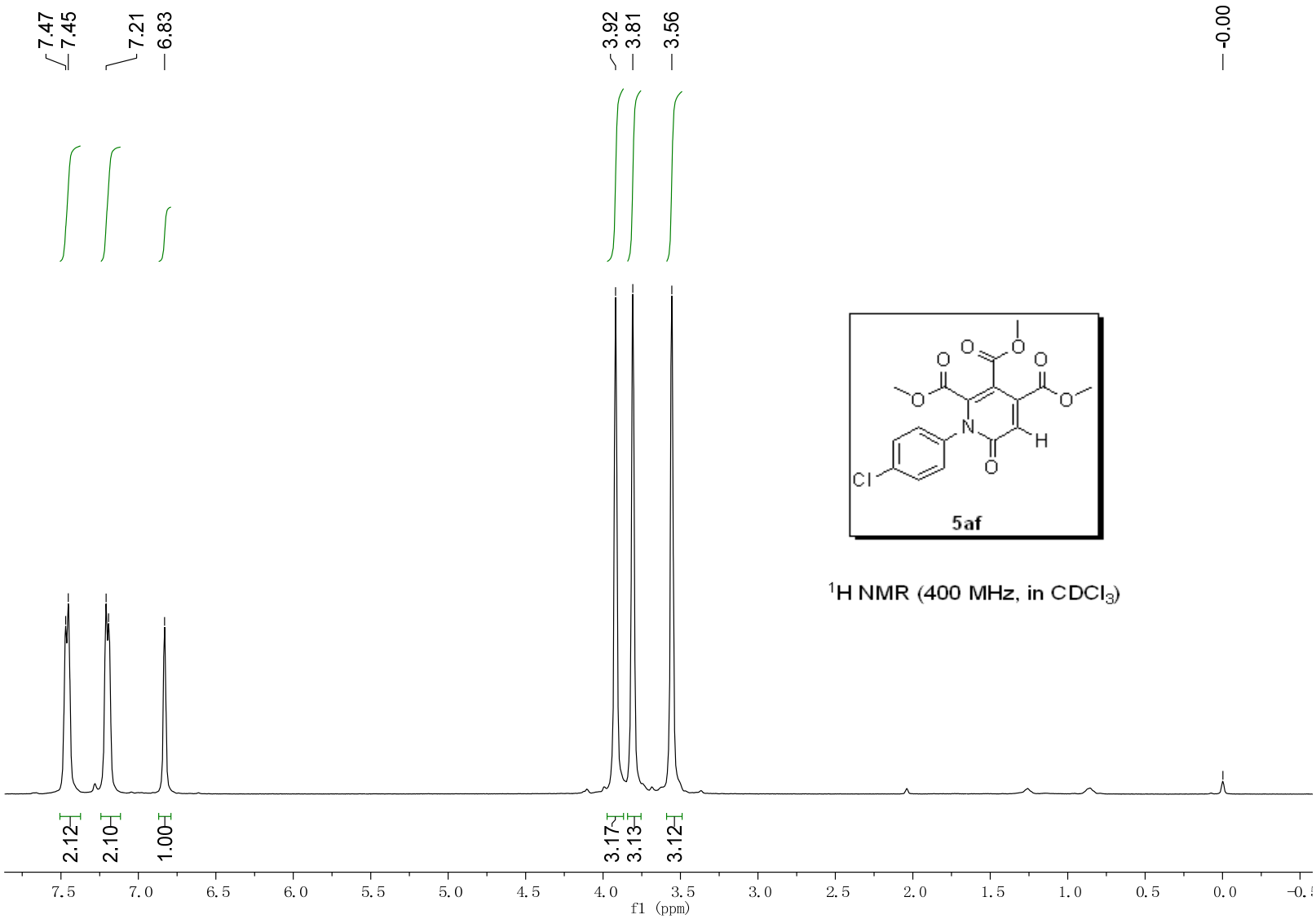


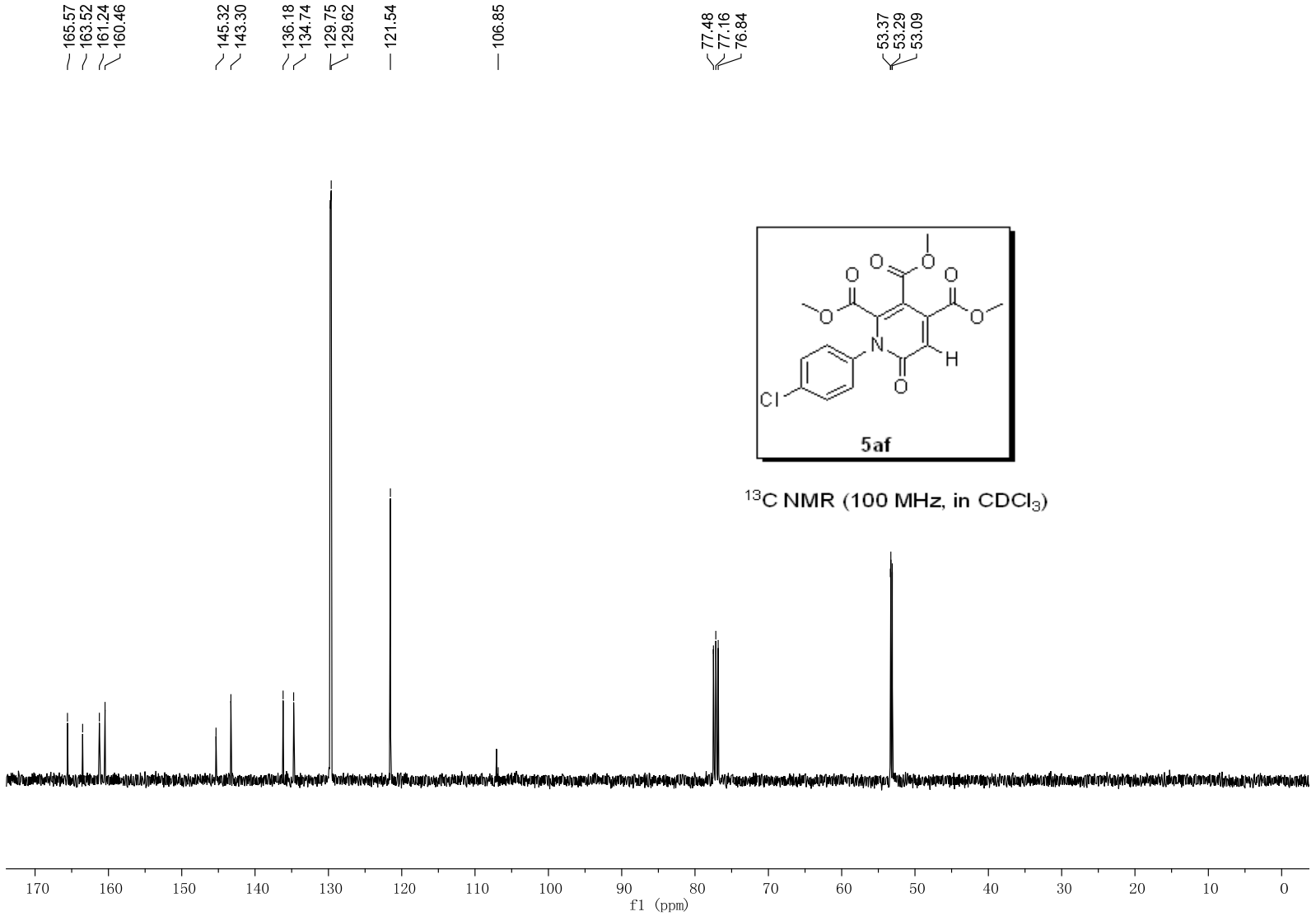


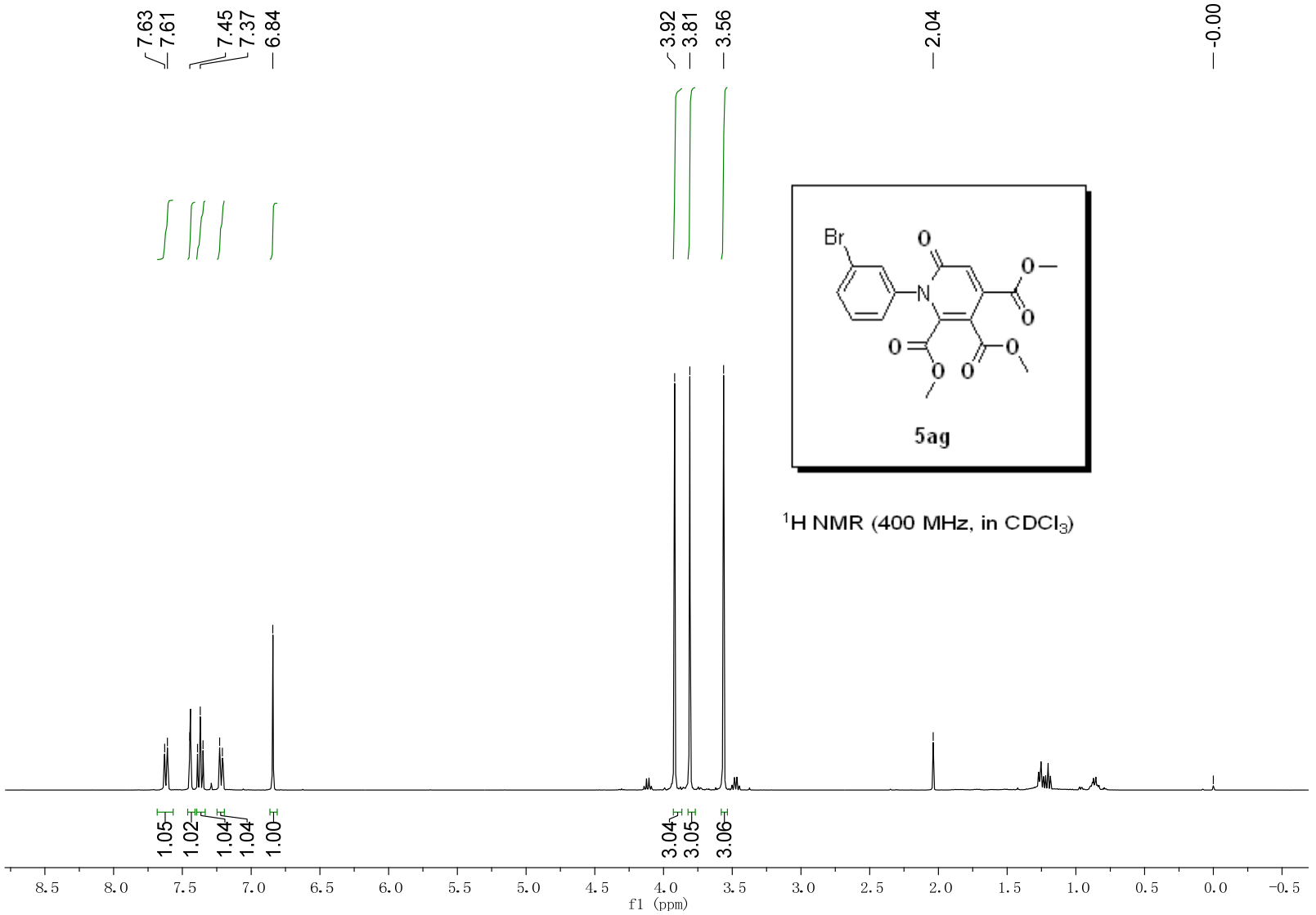


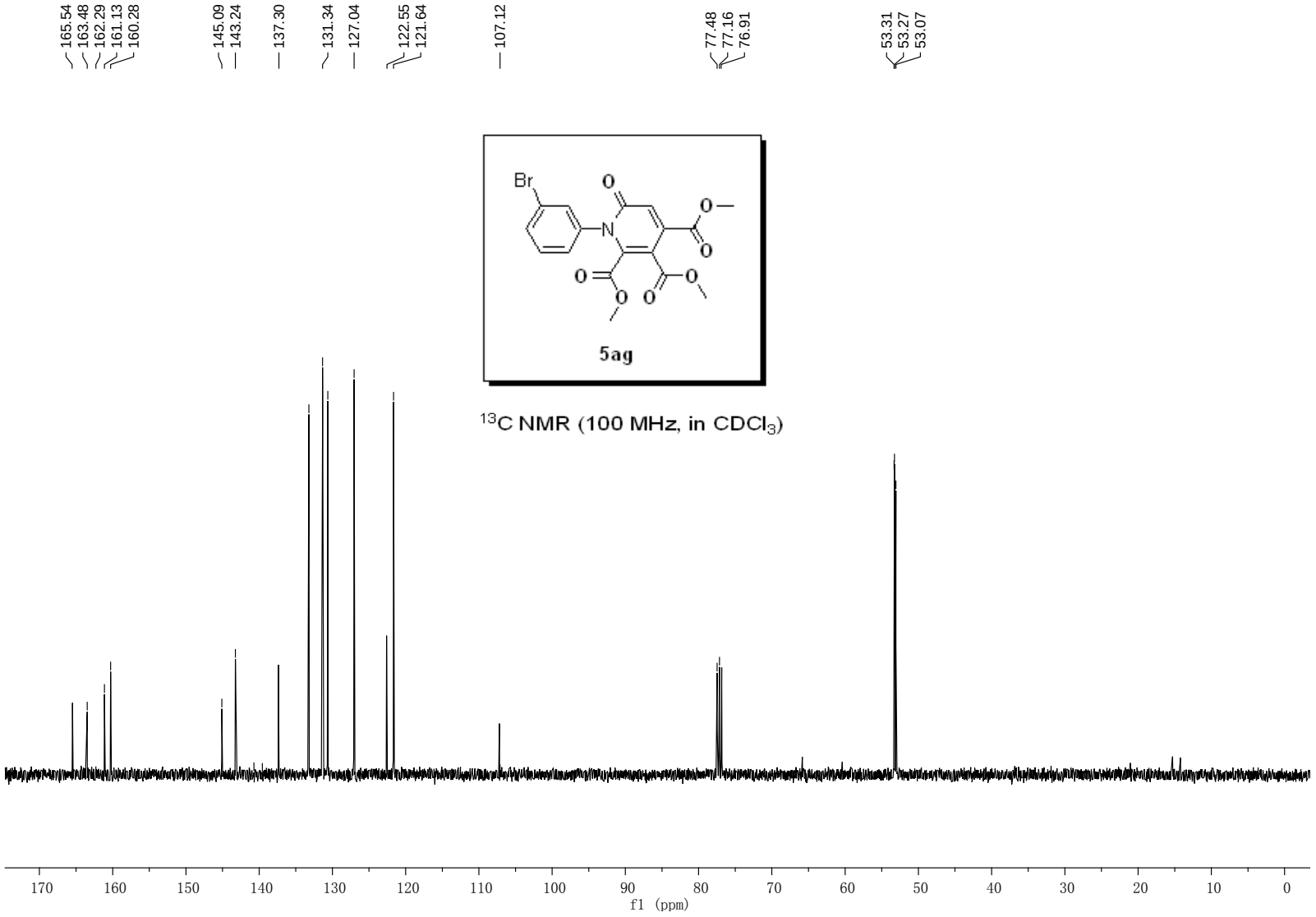


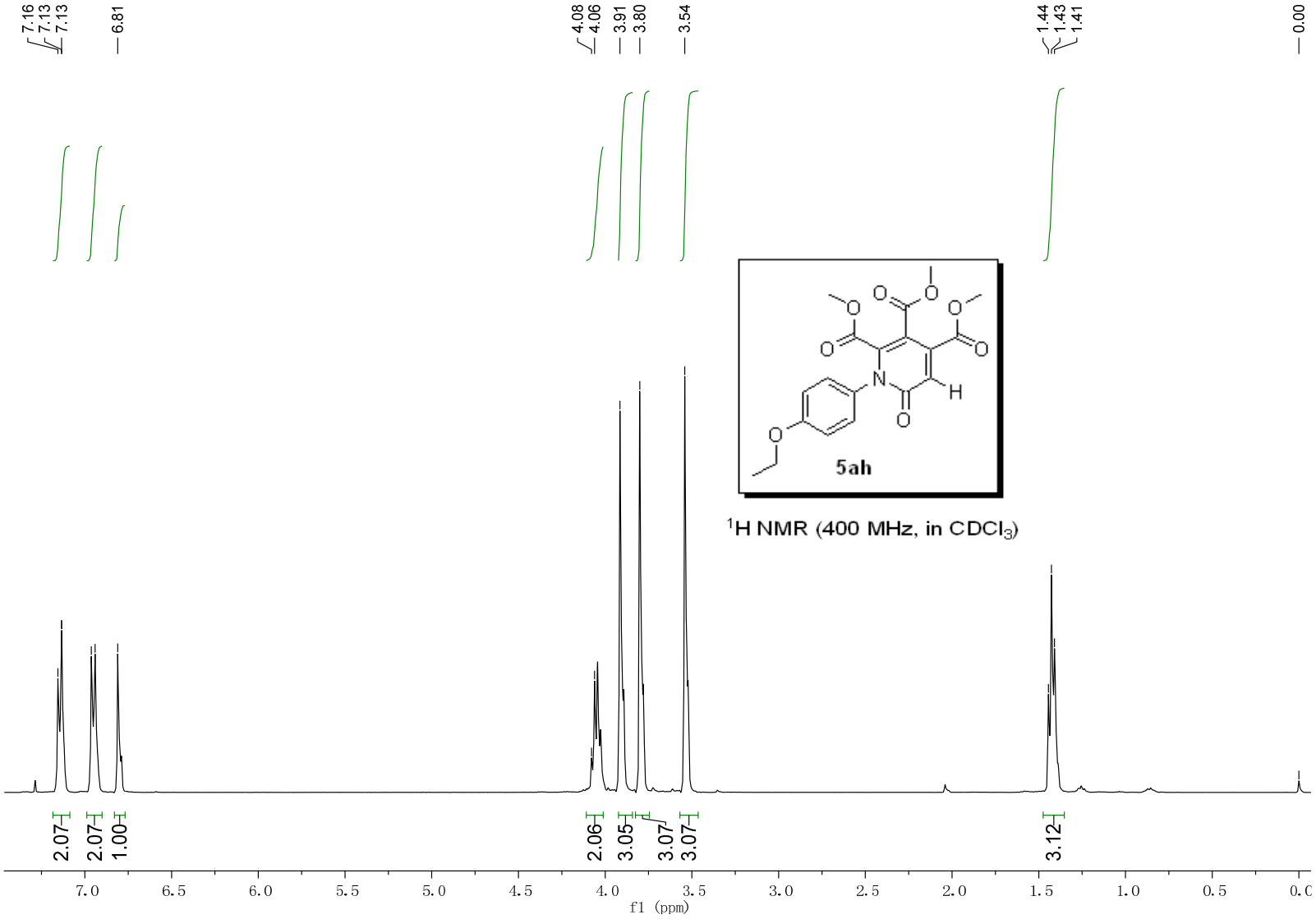


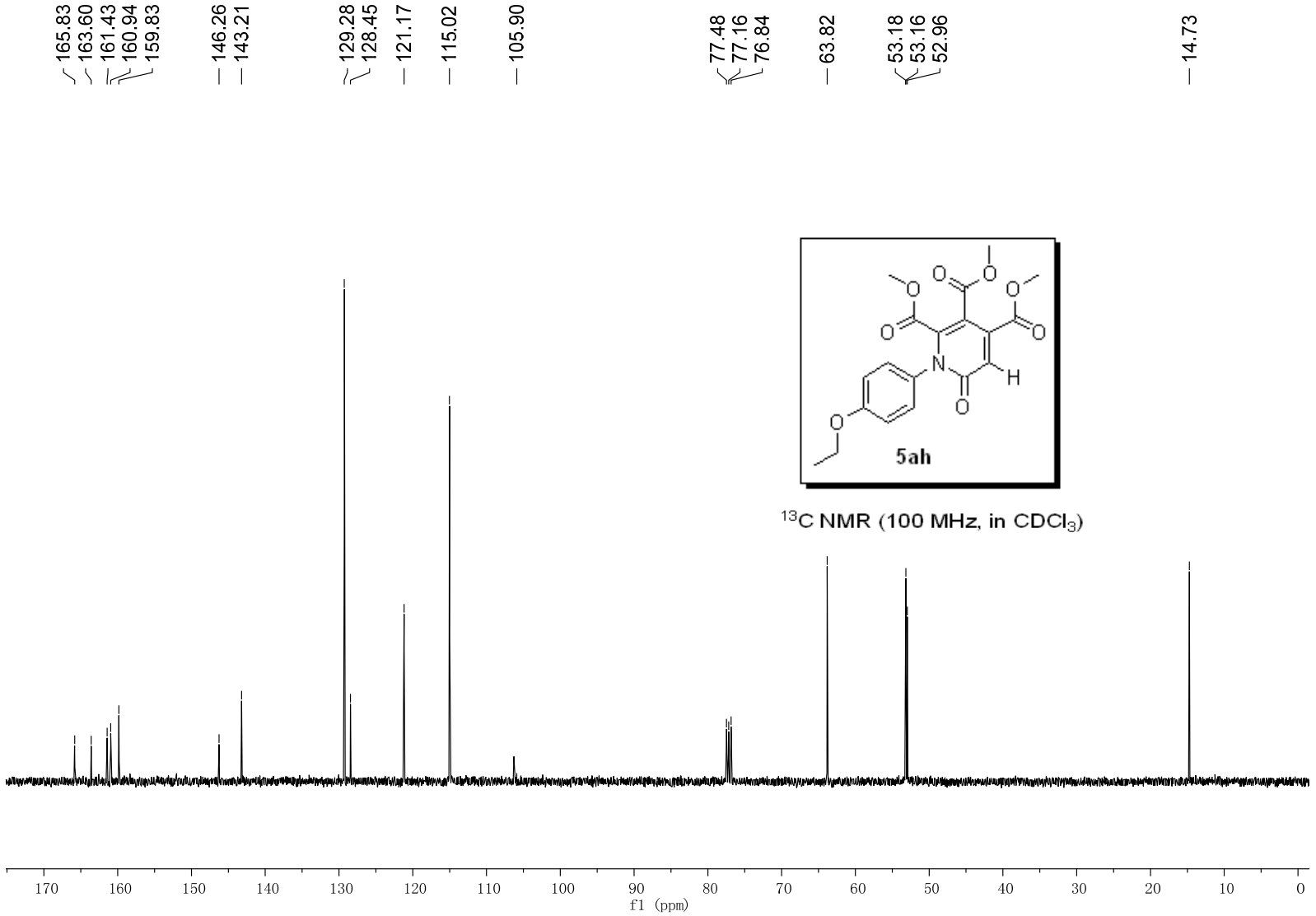


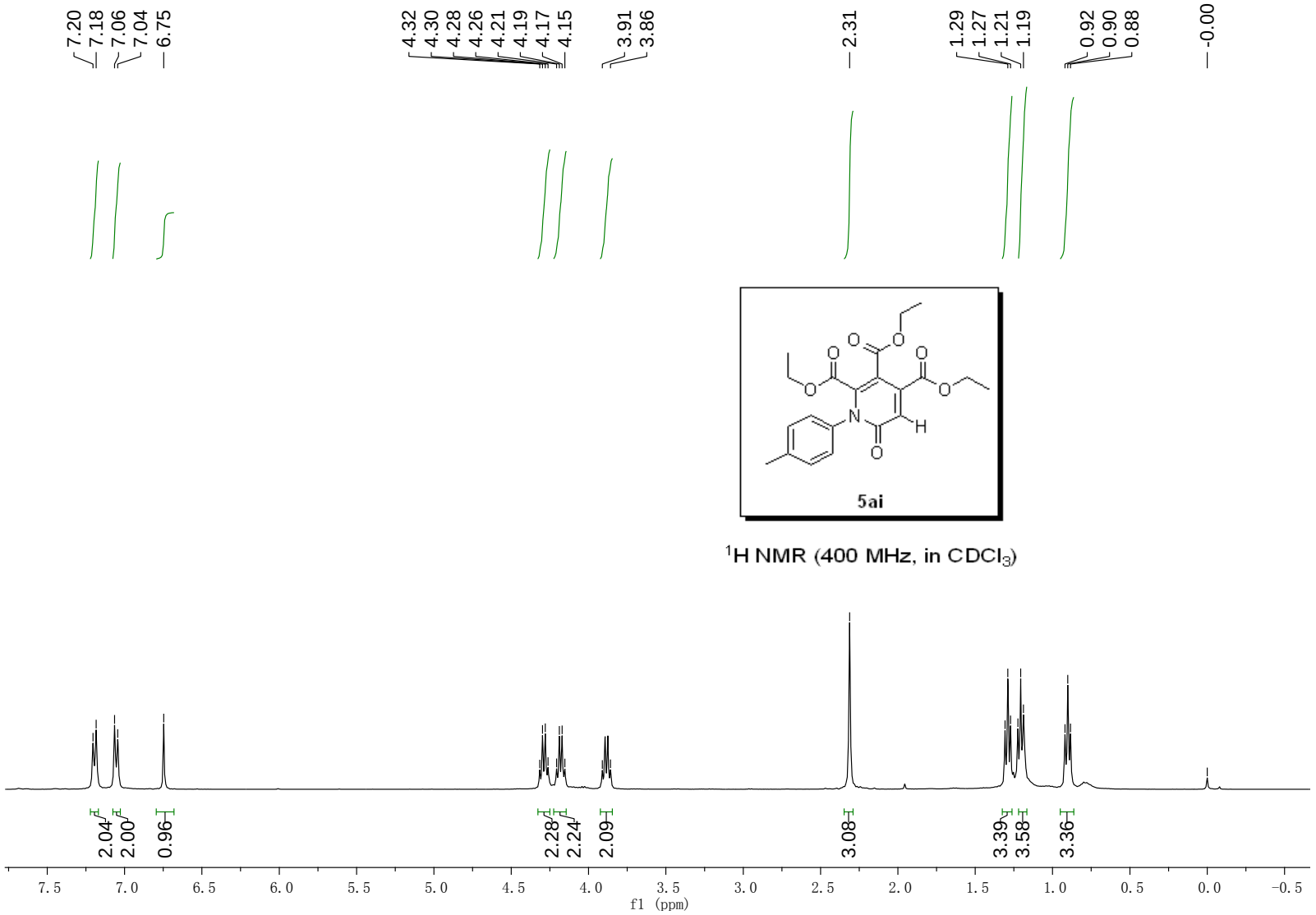




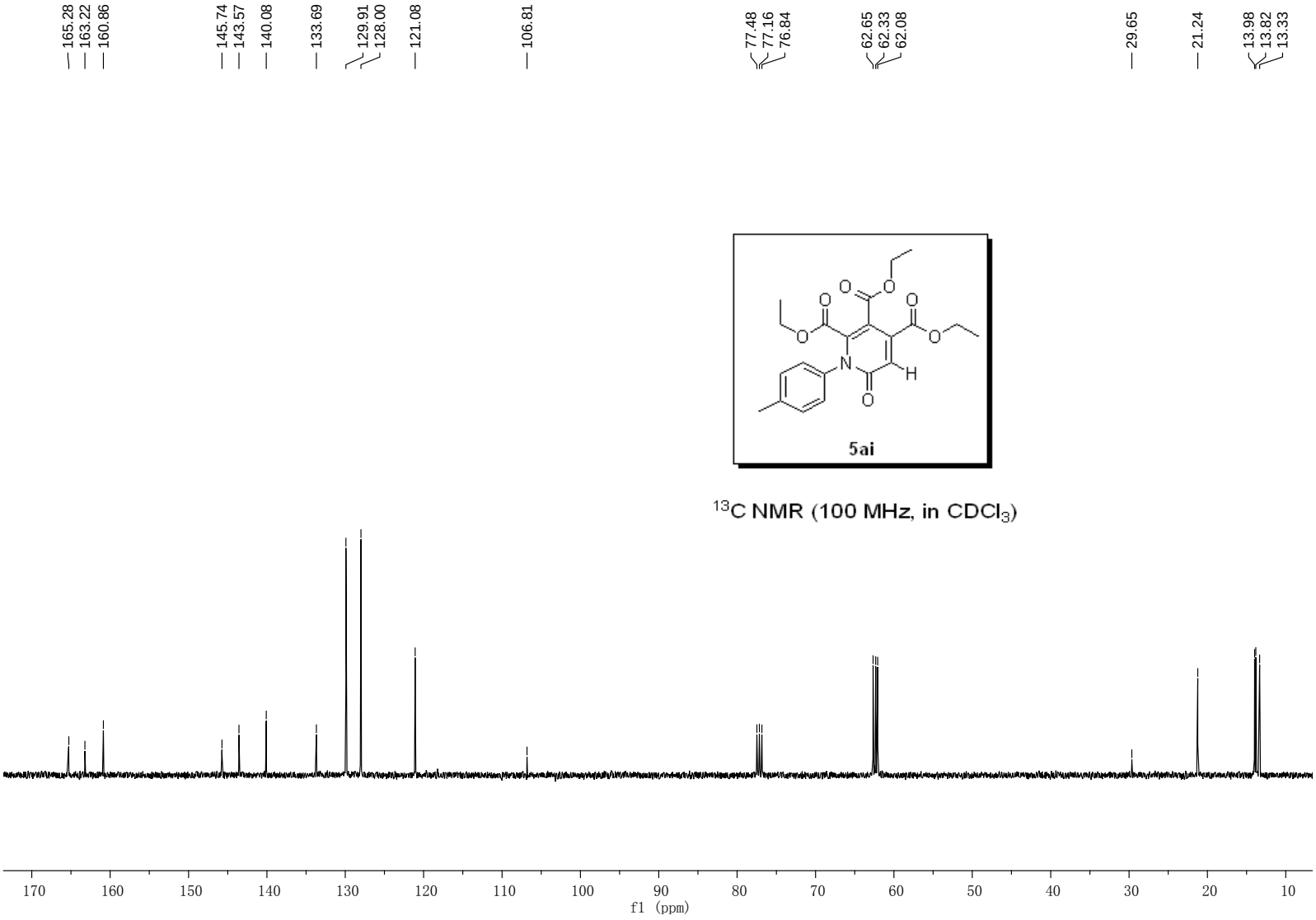


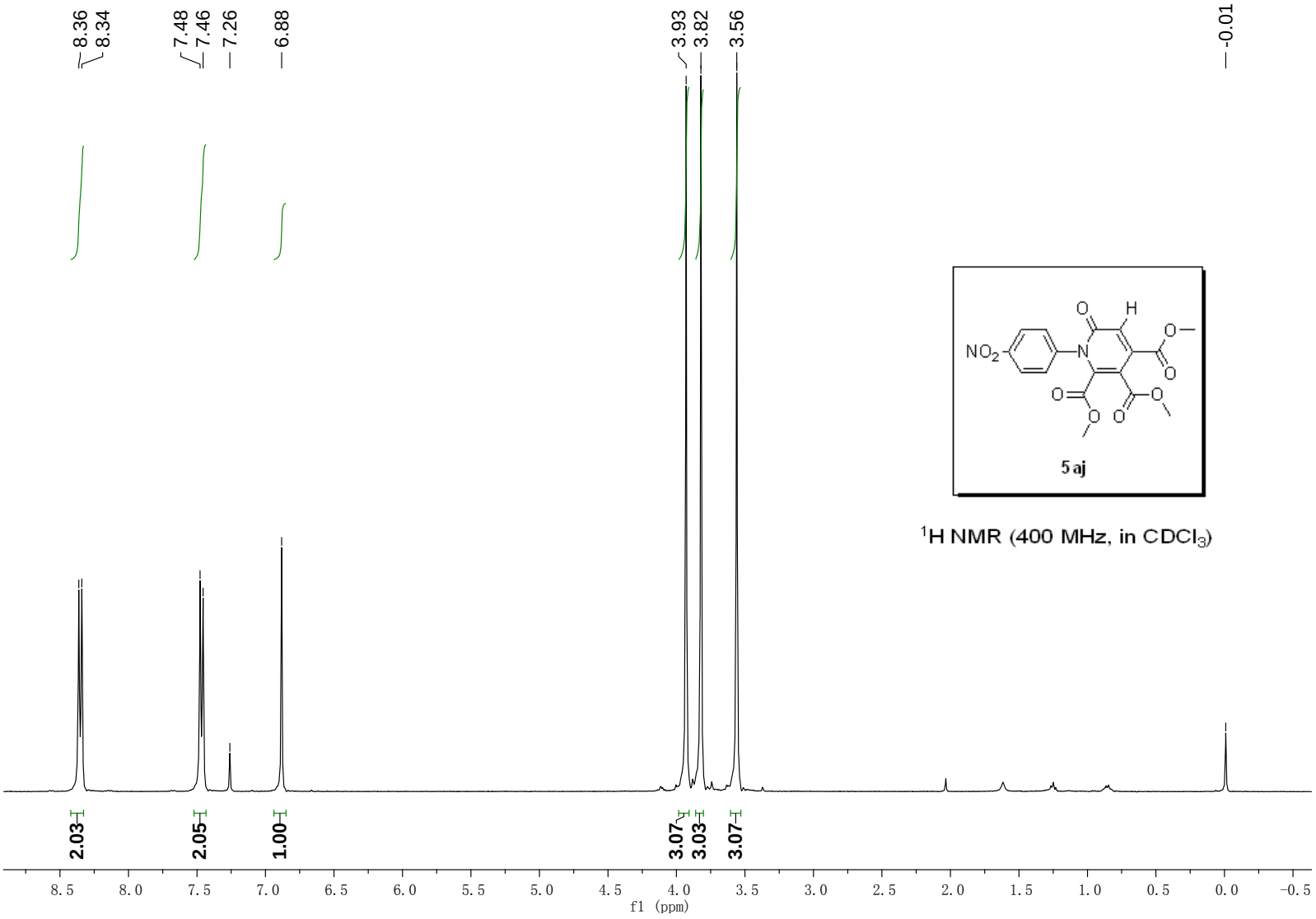


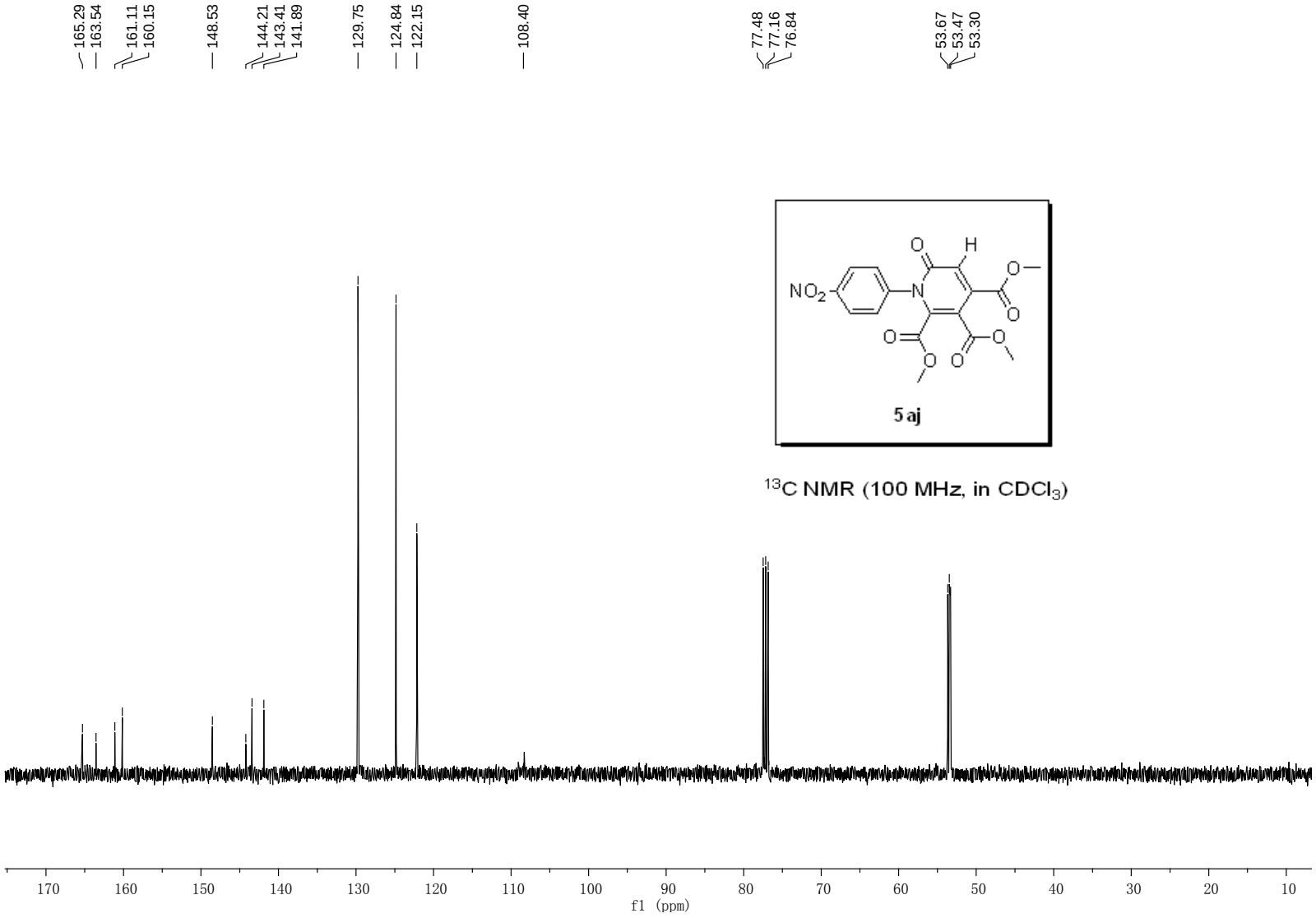


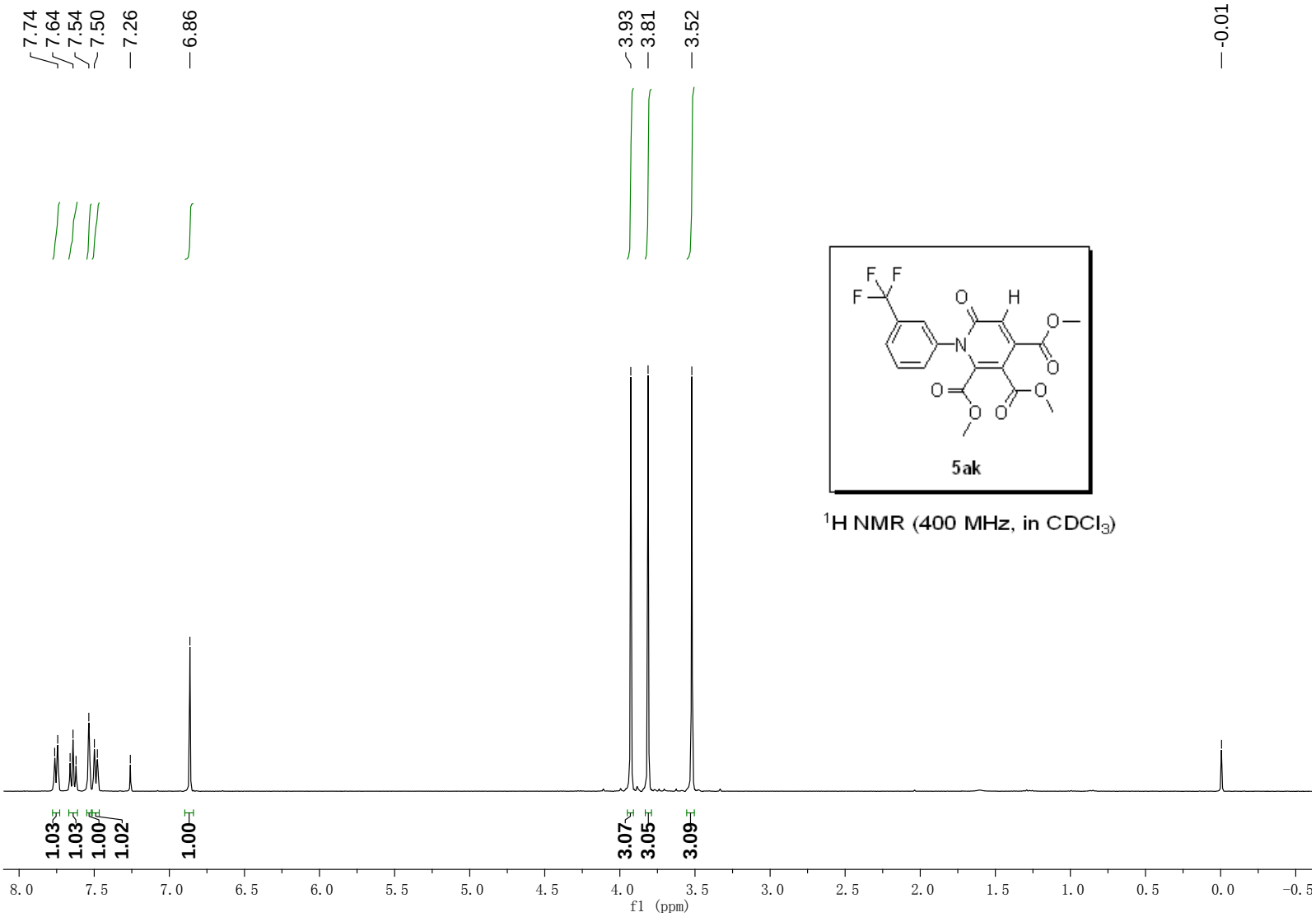


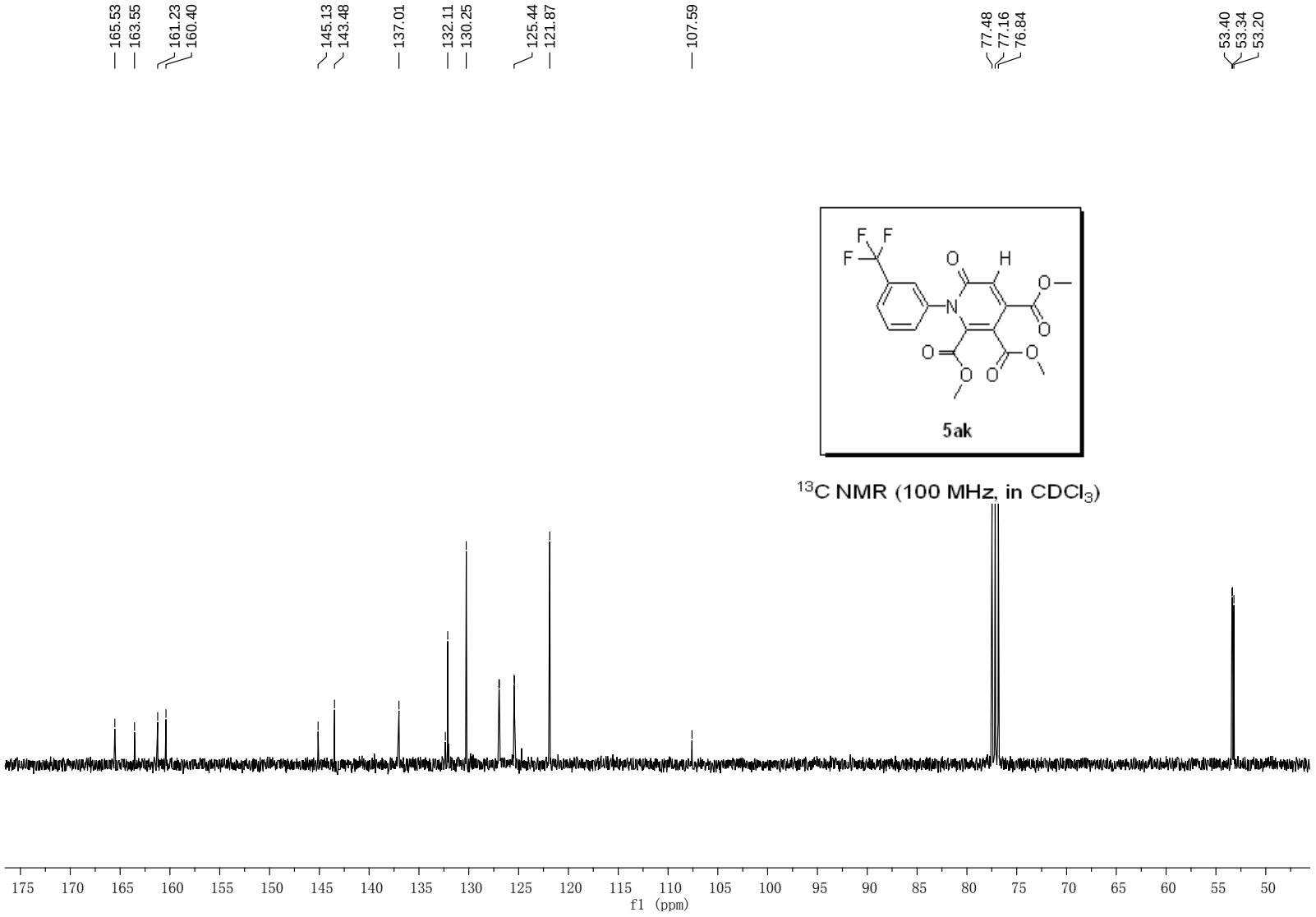
^{1}H NMR (400 MHz, in $CDCl_3$)

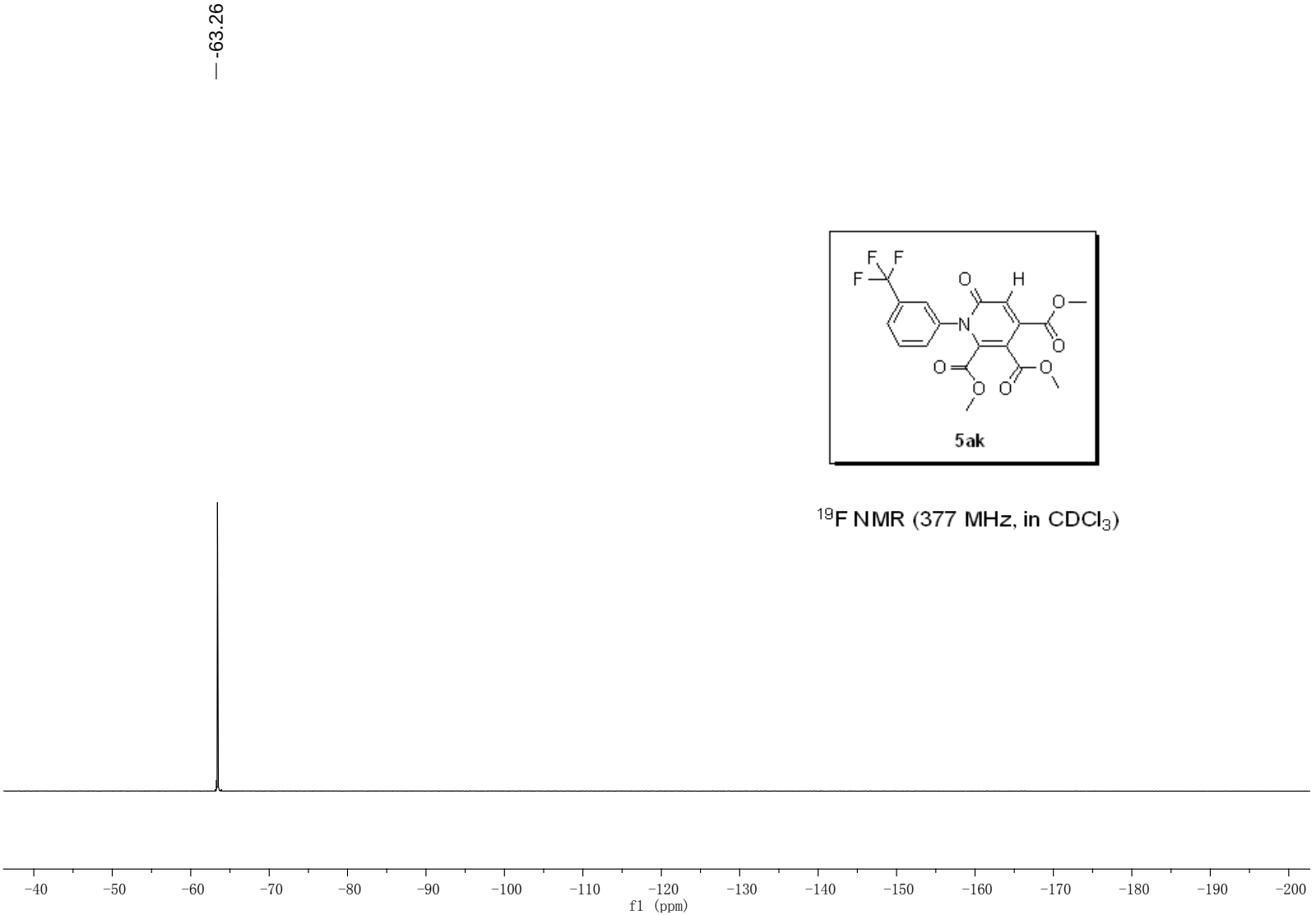




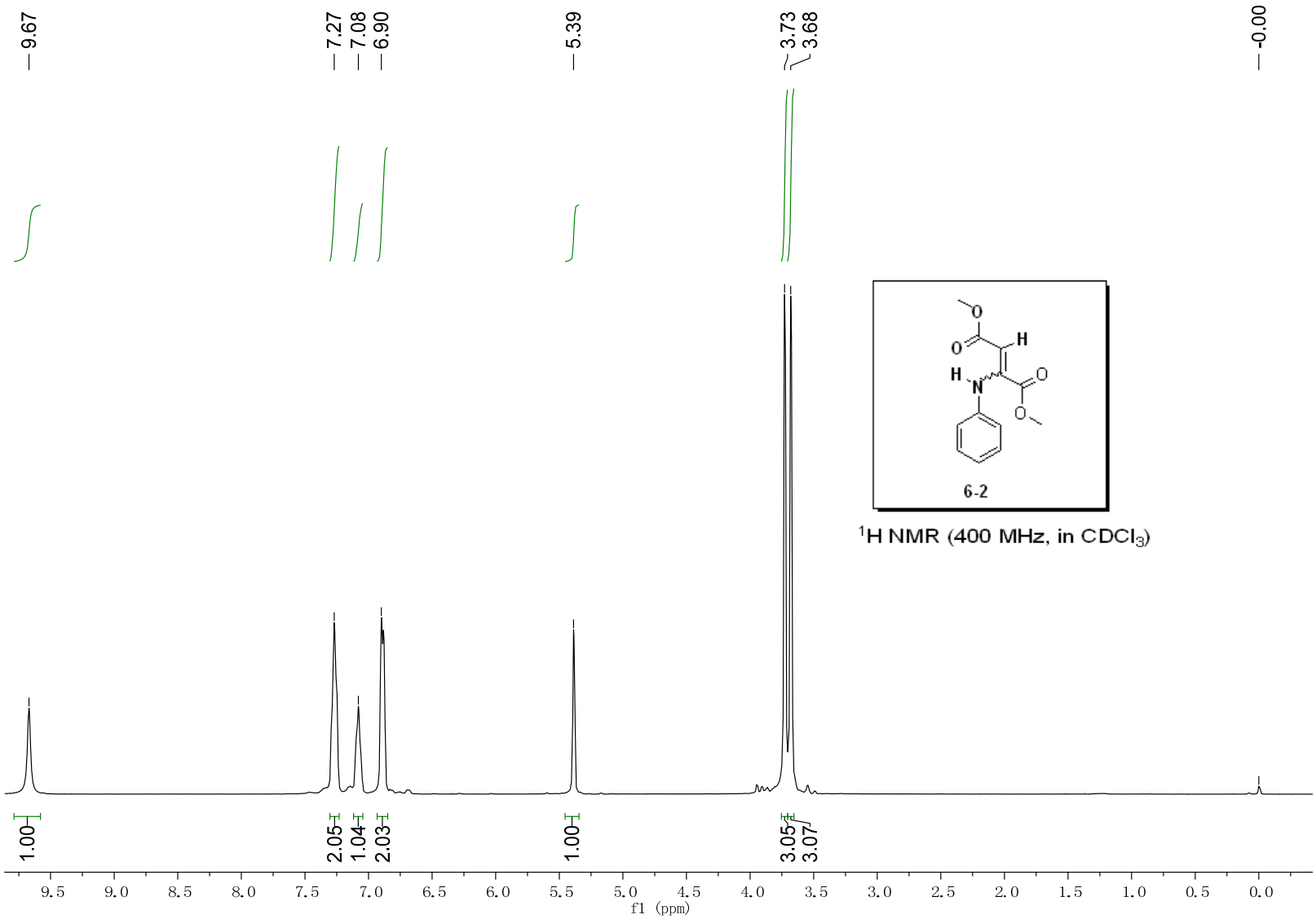


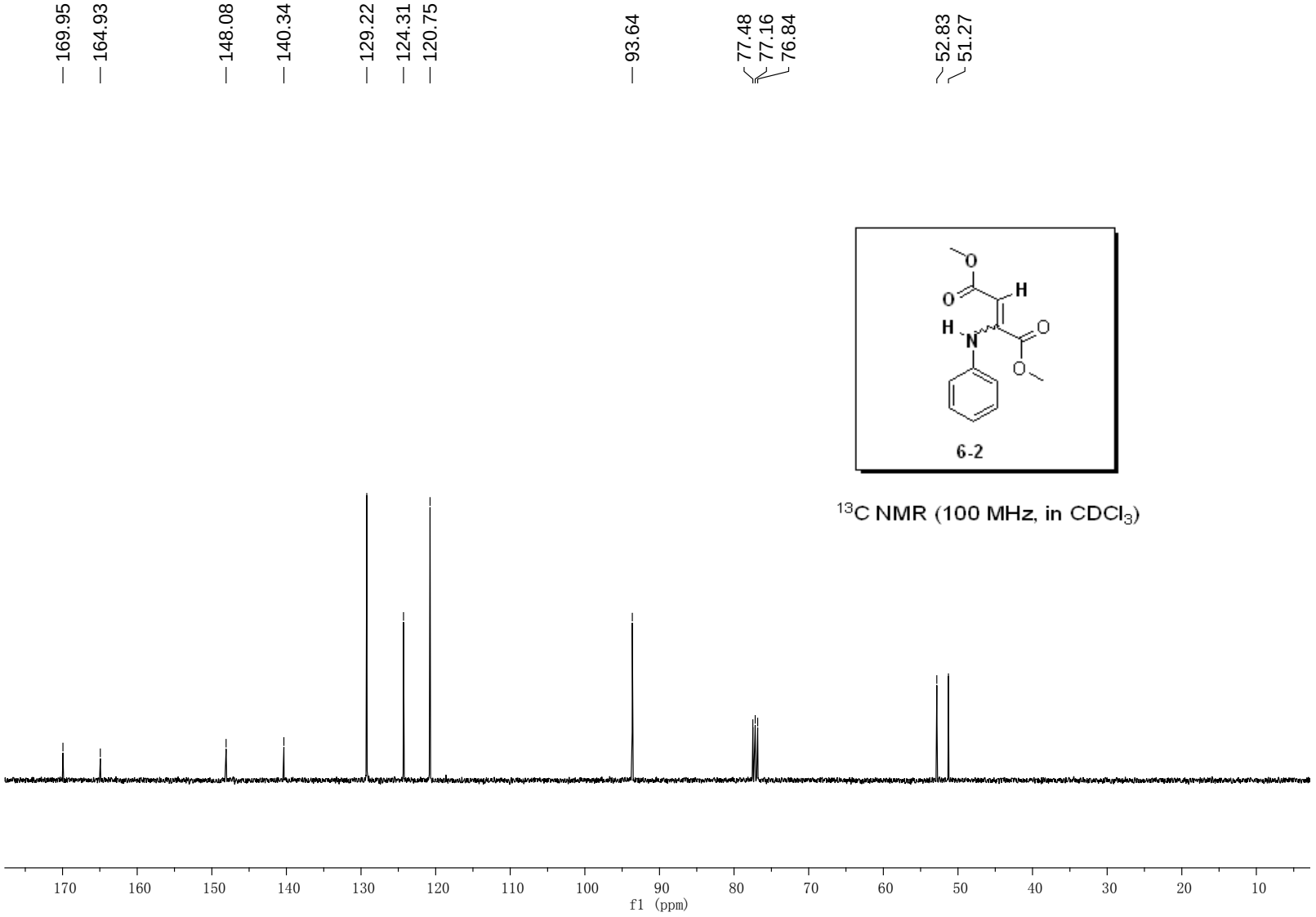


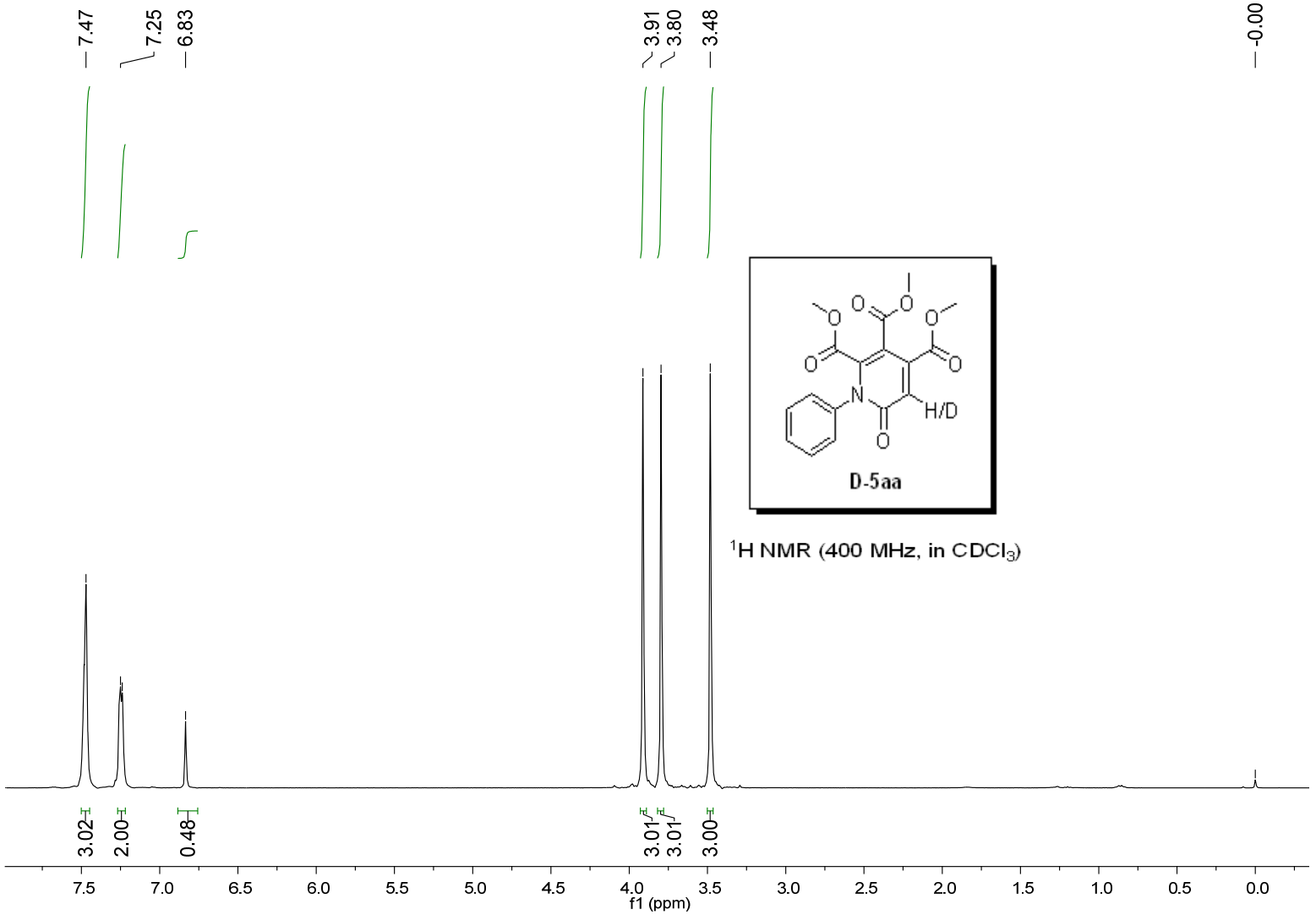


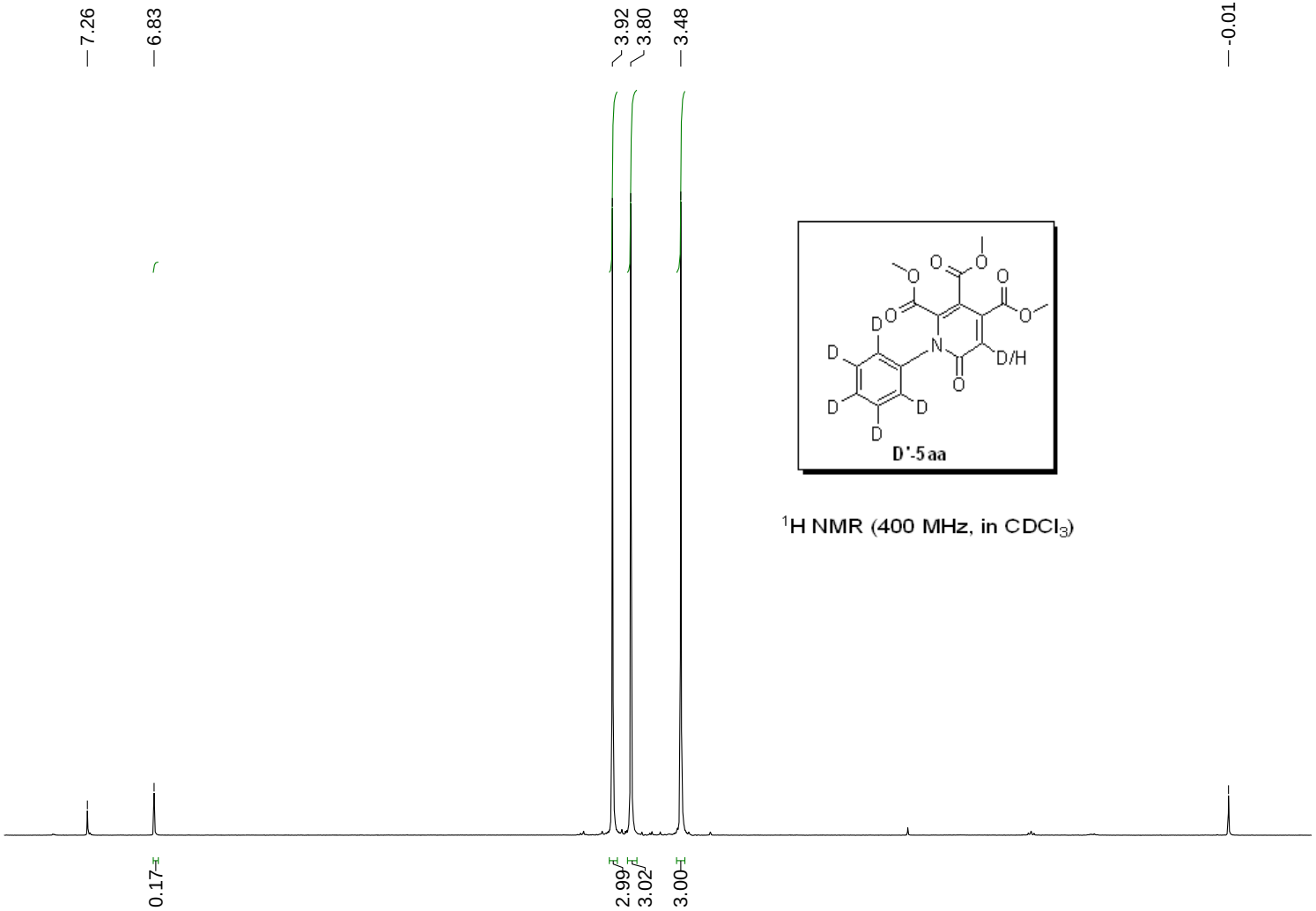


^{19}F NMR (377 MHz, in $CDCl_3$)





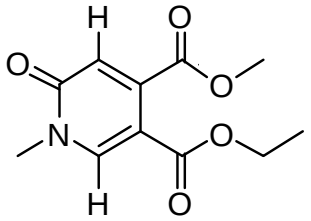




Elemental Composition Report

Single Mass Analysis (displaying only valid results)

Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

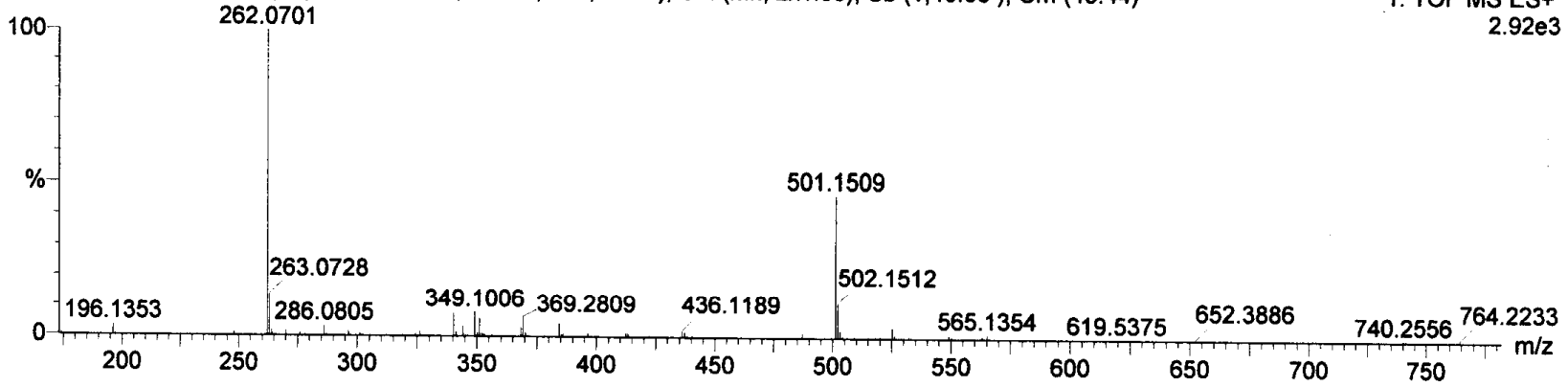


Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100 H: 0-150 N: 1-1 O: 5-5 Na: 1-1

4a

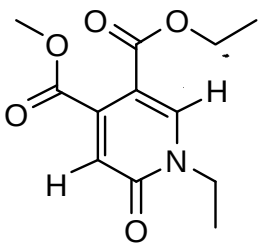
12092637 43 (1.113) AM (Top,4, Ar,5000.0,475.27,1.00,LS 10); Sm (Mn, 2x1.00); Sb (1,40.00); Cm (43:44)

16:00:08
1: TOF MS ES+
2.92e3



Minimum:				-200.0		
Maximum:		5.0	5.0	200.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
262.0701	262.0691	1.0	3.8	5.5	1.2	C11 H13 N O5 Na

Elemental Composition Report



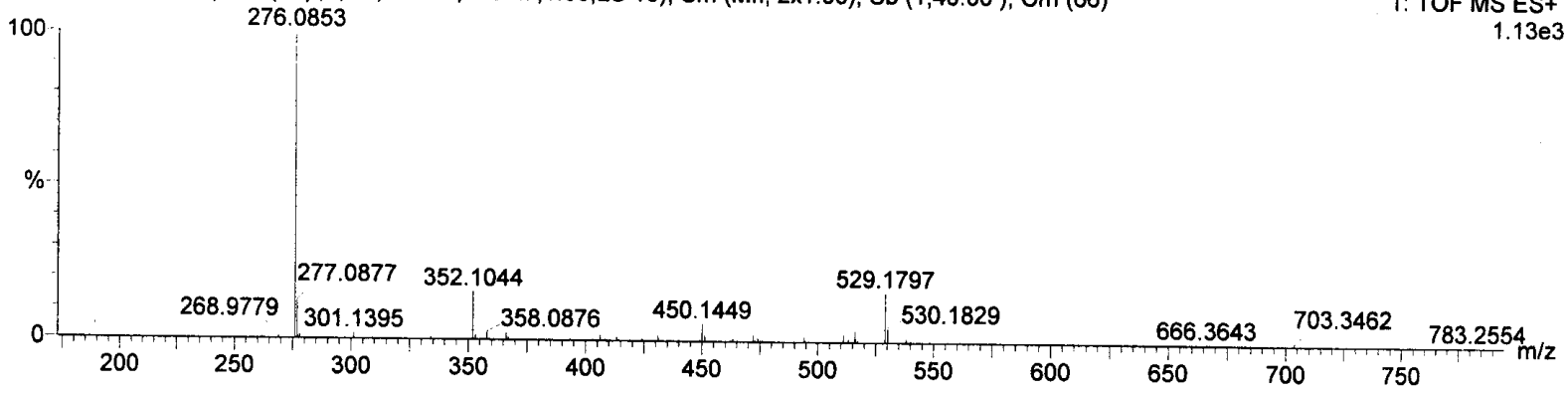
Single Mass Analysis (displaying only valid results)
Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100 H: 0-150 N: 1-1 O: 5-5 Na: 1-1

4ab

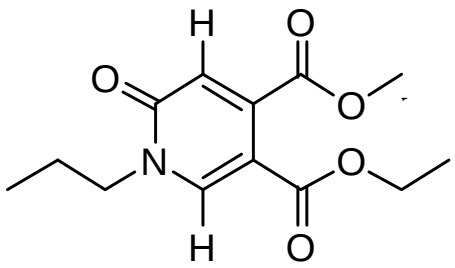
12120303 66 (1.666) AM (Top,4, Ar,5000.0,475.27,1.00,LS 10); Sm (Mn, 2x1.00); Sb (1,40.00); Cm (66)

10:00:11
1: TOF MS ES+
1.13e3



Minimum:				-200.0		
Maximum:		5.0	5.0	200.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
276.0853	276.0848	0.5	1.8	5.5	4.0	C12 H15 N O5 Na

Elemental Composition Report

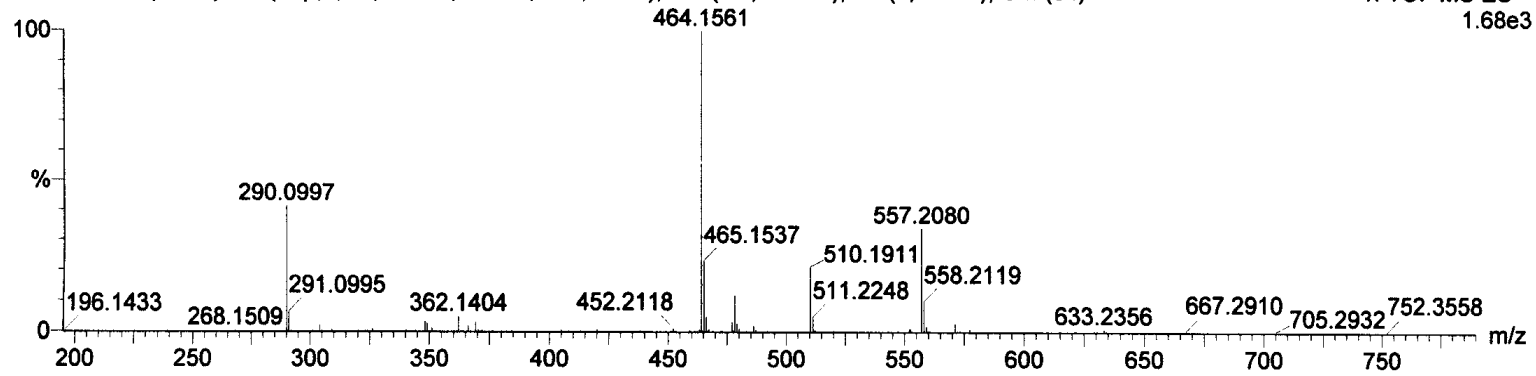


Single Mass Analysis (displaying only valid results)
Tolerance = 50.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100 H: 0-150 N: 1-1 O: 5-5 Na: 1-1

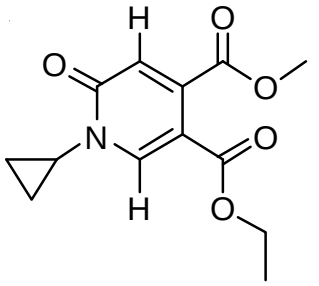
4ac
12092638 54 (1.365) AM (Top,4, Ar,5000.0,475.27,1.00,LS 10); Sm (Mn, 2x1.00); Sb (1,40.00); Cm (54)

16:26:27
1: TOF MS ES+
1.68e3



Minimum:				-200.0		
Maximum:		5.0	50.0	200.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
290.0997	290.1004	-0.7	-2.4	5.5	8.5	C13 H17 N O5 Na

Elemental Composition Report

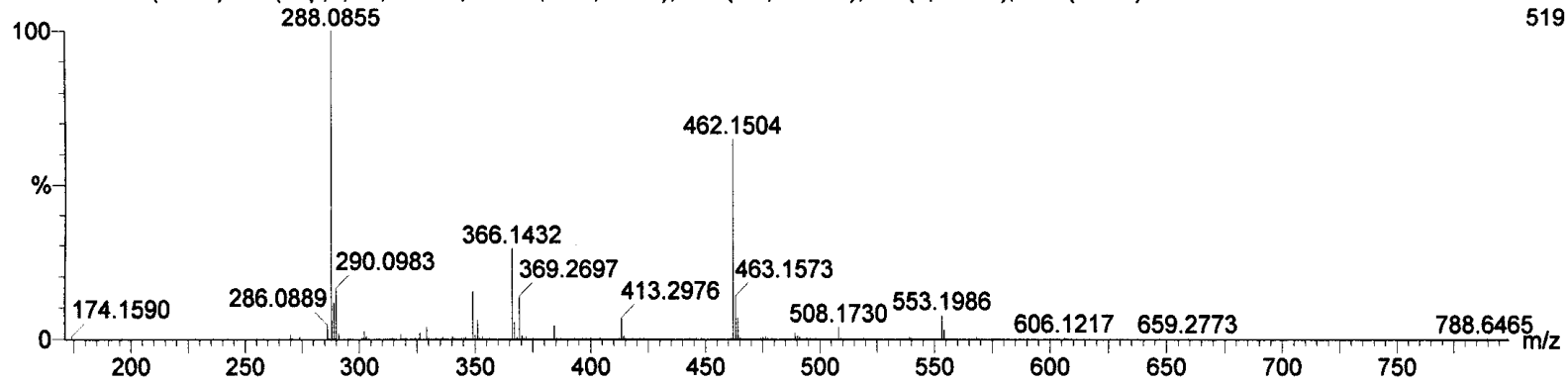


Single Mass Analysis (displaying only valid results)
Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100 H: 0-150 N: 1-1 O: 5-5 Na: 1-1

4ad
12092639 31 (0.810) AM (Top,4, Ar,5000.0,475.27,1.00,LS 10); Sm (Mn, 2x1.00); Sb (1,40.00); Cm (31:32)

16:41:50
1: TOF MS ES+
519

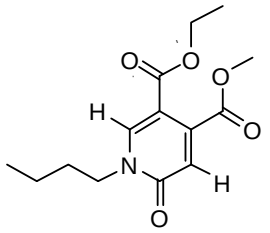


Minimum:				-200.0		
Maximum:		5.0	5.0	200.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
288.0855	288.0848	0.7	2.4	6.5	34.7	C13 H15 N O5 Na

Elemental Composition Report

Single Mass Analysis (displaying only valid results)

Tolerance = 50.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

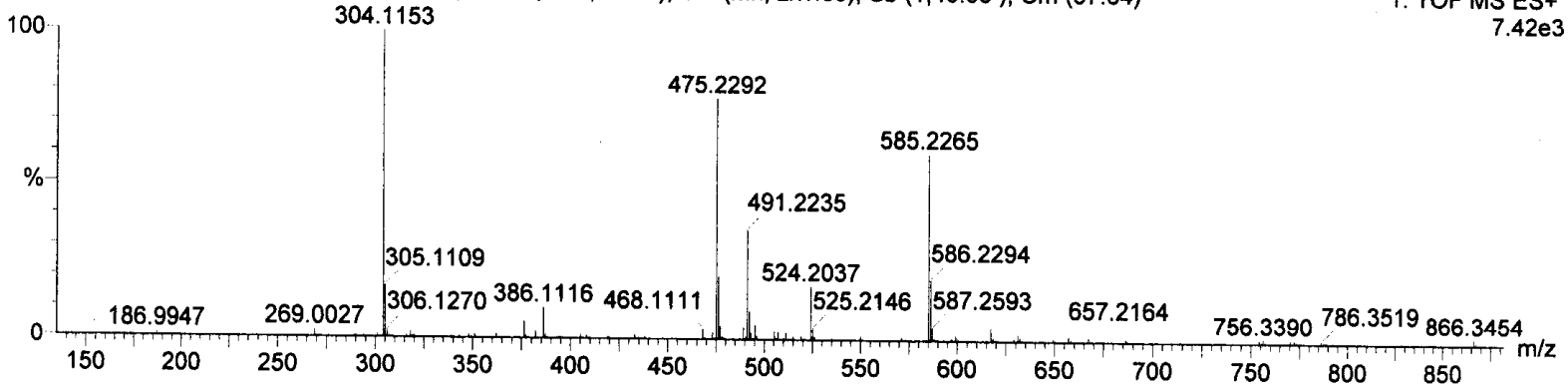


Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100 H: 0-150 N: 1-1 O: 5-5 Na: 1-1

4ae

12120305 58 (1.476) AM (Top,4, Ar,5000.0,475.27,1.00,LS 10); Sm (Mn, 2x1.00); Sb (1,40.00); Cm (57:64)

10:16:24
1: TOF MS ES+
7.42e3

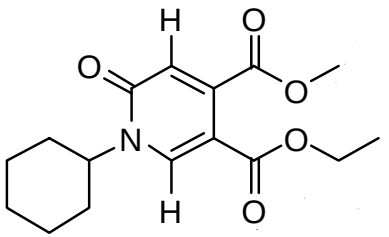


Minimum:				-200.0		
Maximum:		5.0	50.0	200.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
304.1153	304.1161	-0.8	-2.6	5.5	6.2	C14 H19 N O5 Na

Elemental Composition Report

Single Mass Analysis (displaying only valid results)

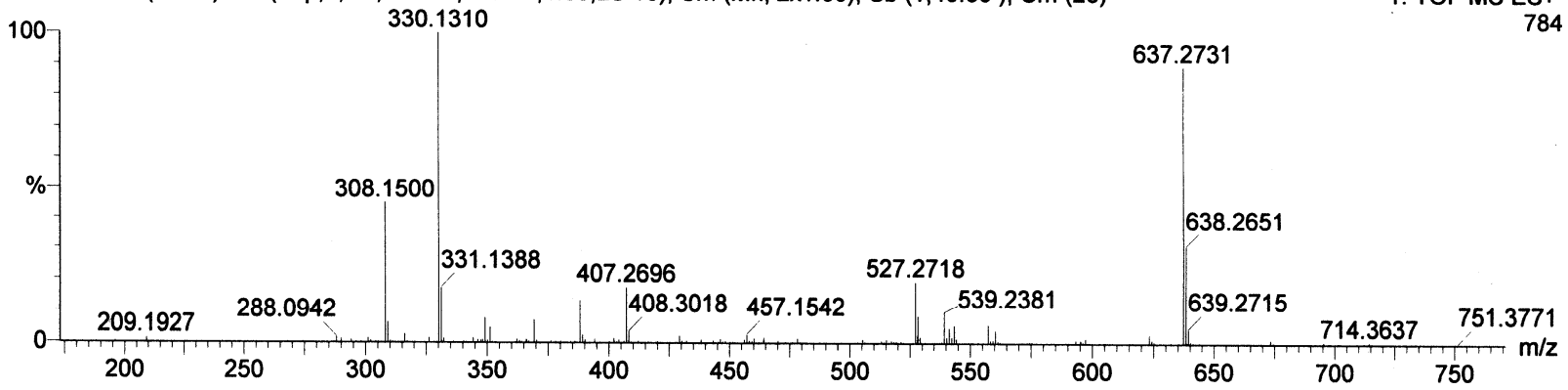
Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None



Monoisotopic Mass, Even Electron Ions
12 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100 H: 0-150 N: 1-1 O: 5-5 Na: 1-1

4a
12092640 26 (0.675) AM (Top,4, Ar,5000.0,475.27,1.00,LS 10); Sm (Mn, 2x1.00); Sb (1,40.00); Cm (26)

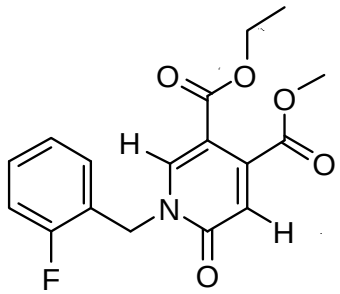
16:58:56
1: TOF MS ES+
784



Minimum:				-200.0		
Maximum:		5.0	5.0	200.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
330.1310	330.1317	-0.7	-2.1	6.5	7.9	C16 H21 N O5 Na

Elemental Composition Report

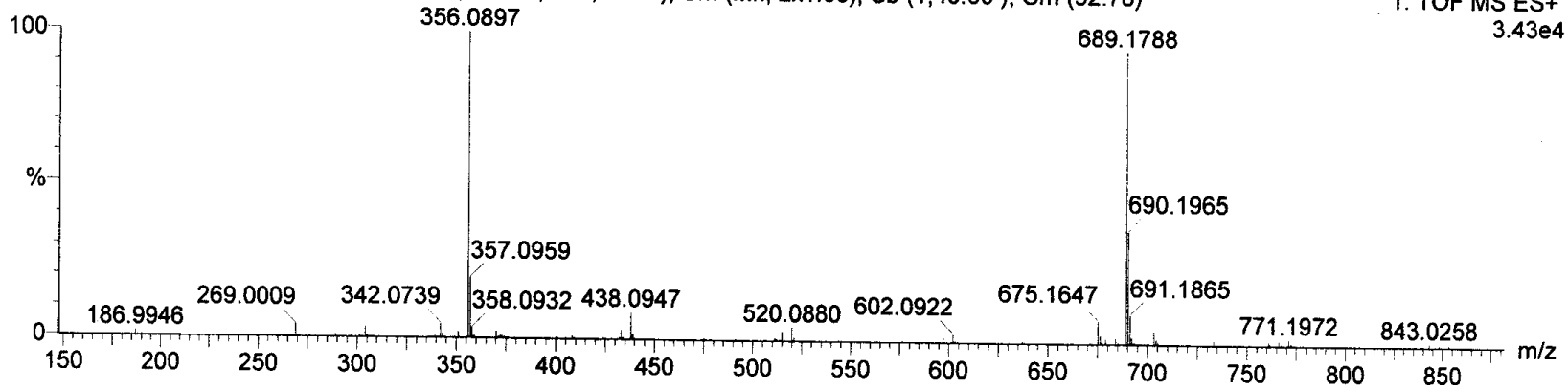
Single Mass Analysis (displaying only valid results)
Tolerance = 50.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None



Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100 H: 0-150 N: 1-1 O: 5-5 F: 1-1 Na: 1-1

4 a g
12120306 53 (1.338) AM (Top,4, Ar,5000.0,475.27,1.00,LS 10); Sm (Mn, 2x1.00); Sb (1,40.00); Cm (52:78)

10:23:04
1: TOF MS ES+
3.43e4

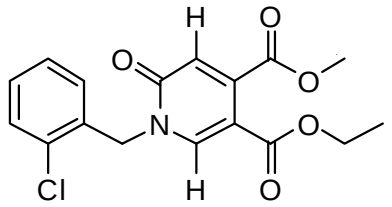


Minimum:				-200.0		
Maximum:		5.0	50.0	200.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
356.0897	356.0910	-1.3	-3.7	9.5	14.3	C17 H16 N O5 F Na

Elemental Composition Report

Single Mass Analysis (displaying only valid results)

Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

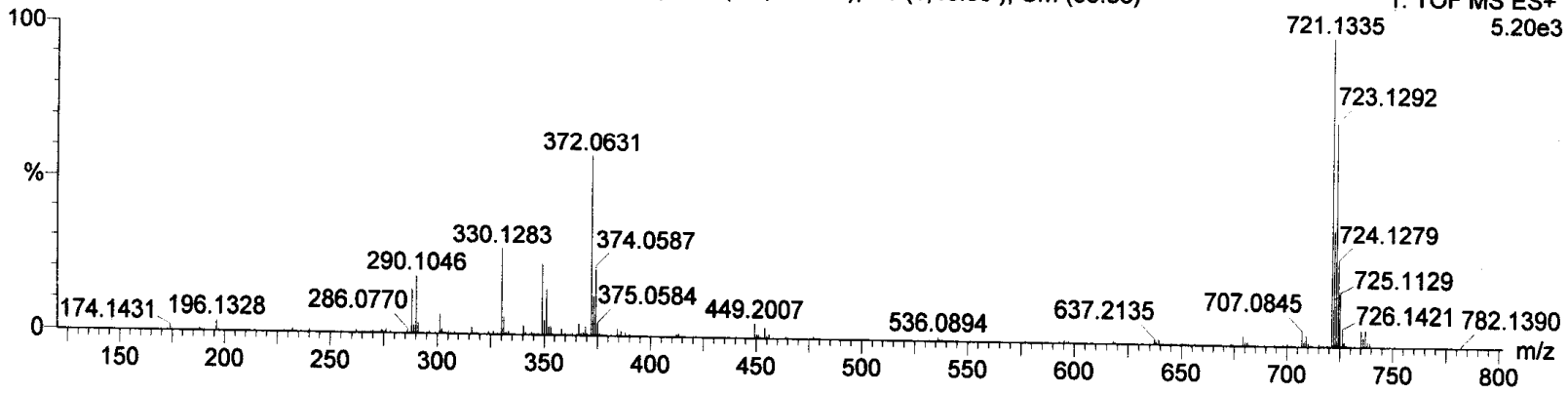


Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100 H: 0-150 N: 1-1 O: 5-5 Na: 1-1 Cl: 1-1

4a

12092641 35 (0.881) AM (Top,4, Ar,5000.0,475.27,1.00,LS 10); Sm (Mn, 2x1.00); Sb (1,40.00); Cm (35:38)

17:09:57
1: TOF MS ES+
5.20e3

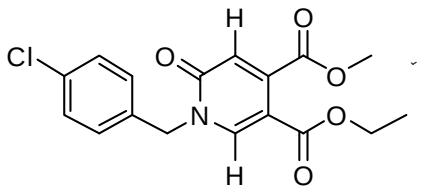


Minimum:				-200.0		
Maximum:		5.0	5.0	200.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
372.0631	372.0615	1.6	4.3	9.5	2.1	C17 H16 N O5 Na Cl

Elemental Composition Report

Single Mass Analysis (displaying only valid results)

Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

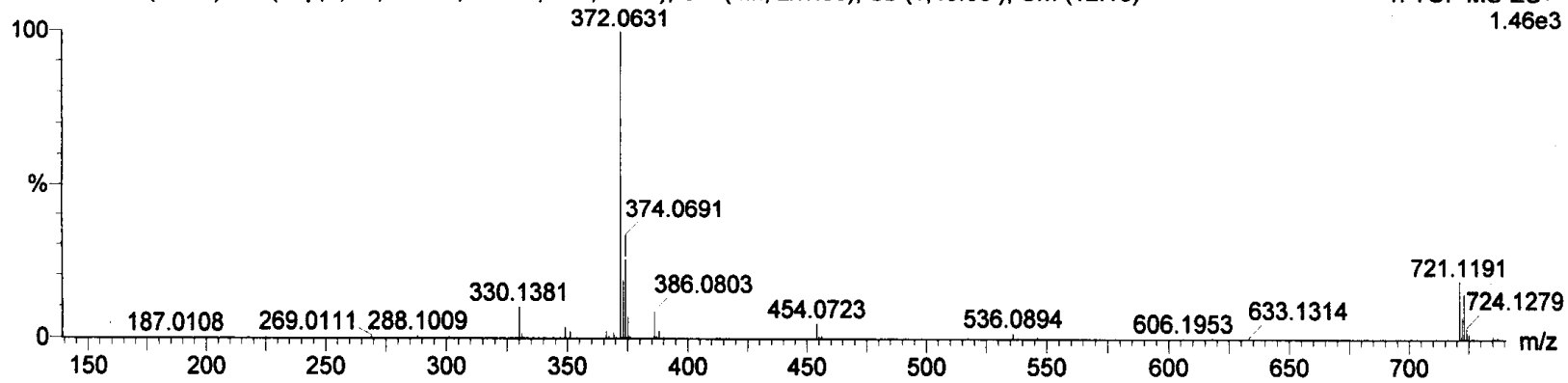


Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100 H: 0-150 N: 1-1 O: 5-5 Na: 1-1 Cl: 1-1

4a

12092642 12 (0.289) AM (Top,4, Ar,5000.0,475.27,1.00,LS 10); Sm (Mn, 2x1.00); Sb (1,40.00); Cm (12:13)

17:19:26
1: TOF MS ES+
1.46e3

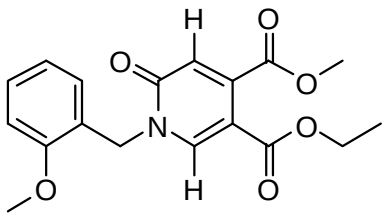


Minimum:				-200.0		
Maximum:		5.0	5.0	200.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
372.0631	372.0615	1.6	4.3	9.5	2.1	C17 H16 N O5 Na Cl

Elemental Composition Report

Single Mass Analysis (displaying only valid results)

Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

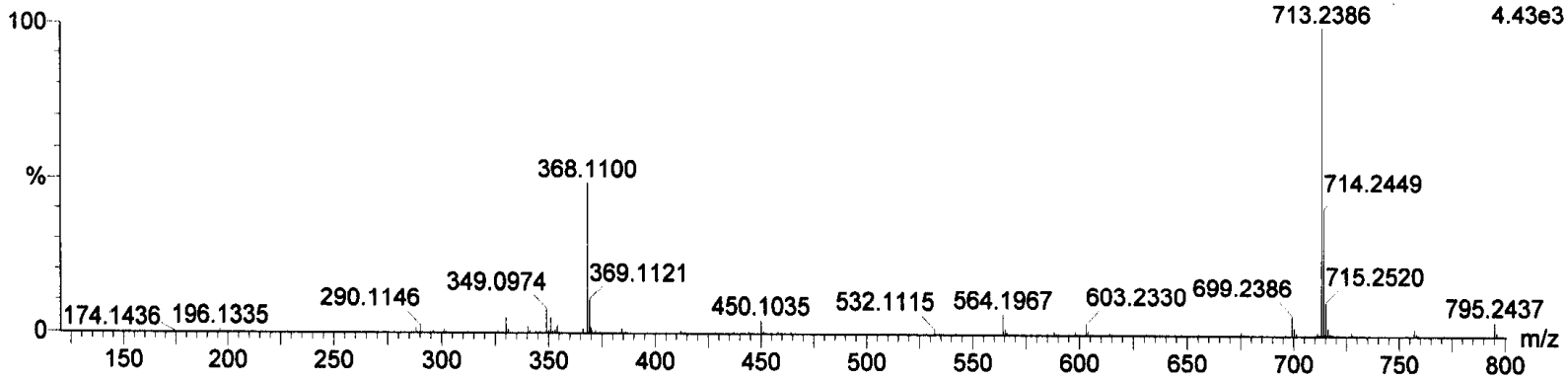


Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100 H: 0-150 N: 1-1 O: 6-6 Na: 1-1

4a

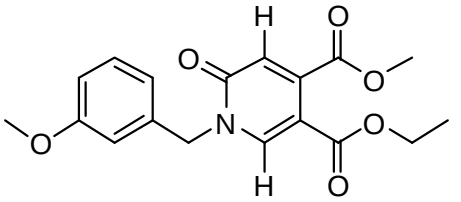
12092643 56 (1.450) AM (Top,4, Ar,5000.0,475.27,1.00,LS 10); Sm (Mn, 2x1.00); Sb (1,40.00); Cm (55:56)

17:27:39
1: TOF MS ES+
4.43e3



Minimum:				-200.0		
Maximum:		5.0	5.0	200.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
368.1100	368.1110	-1.0	-2.7	9.5	1.3	C18 H19 N O6 Na

Elemental Composition Report



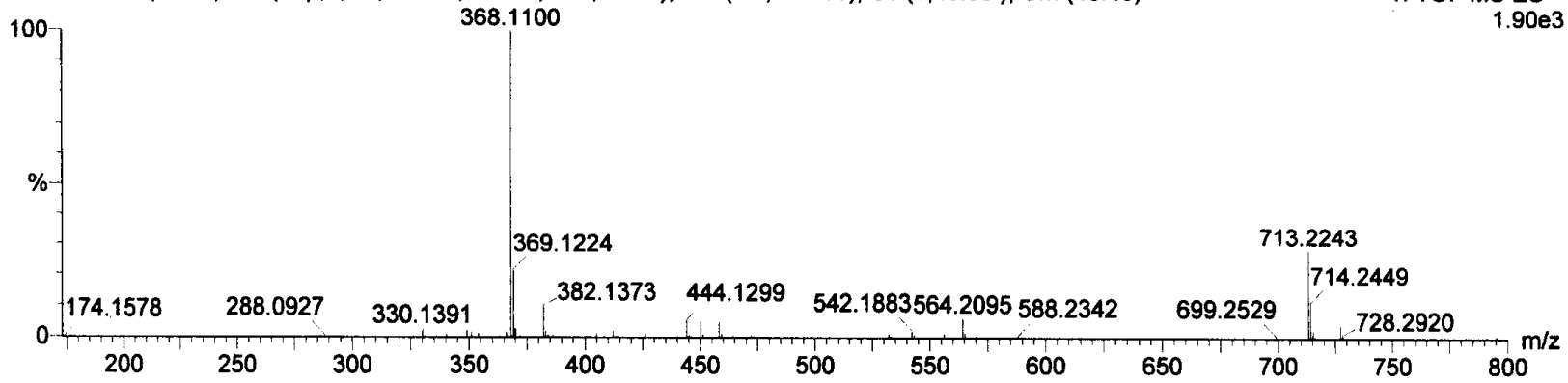
Single Mass Analysis (displaying only valid results)

Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100 H: 0-150 N: 1-1 O: 6-6 Na: 1-1

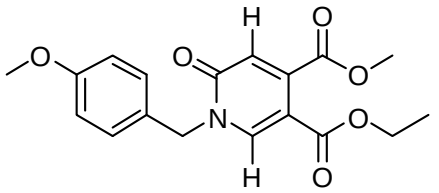
4aK
12092644 48 (1.205) AM (Top,4, Ar,5000.0,475.27,1.00,LS 10); Sm (Mn, 2x1.00); Sb (1,40.00); Cm (48:49)

17:35:45
1: TOF MS ES+
1.90e3



Minimum:				-200.0		
Maximum:		5.0	5.0	200.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
368.1100	368.1110	-1.0	-2.7	9.5	2.4	C18 H19 N O6 Na

Elemental Composition Report



Single Mass Analysis (displaying only valid results)

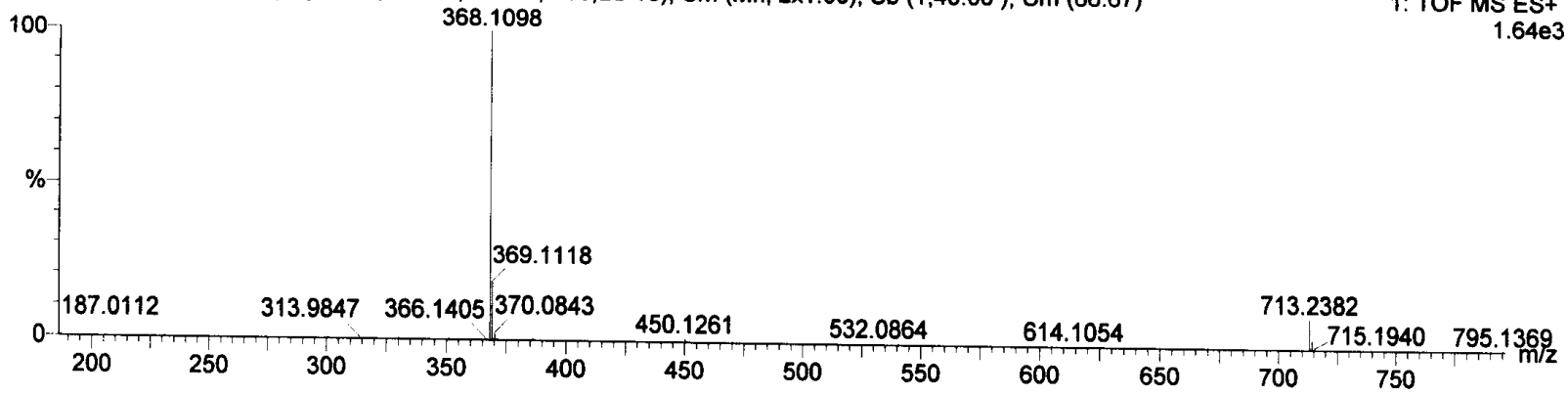
Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100 H: 0-150 N: 1-1 O: 6-6 Na: 1-1

4a

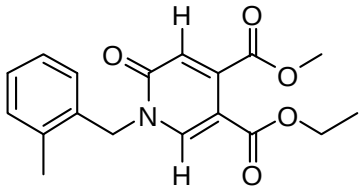
12092645 67 (1.741) AM (Top,4, Ar,5000.0,475.27,1.00,LS 10); Sm (Mn, 2x1.00); Sb (1,40.00); Cm (66:67)

17:50:38
1: TOF MS ES+
1.64e3



Minimum:				-200.0		
Maximum:		5.0	5.0	200.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
368.1098	368.1110	-1.2	-3.3	9.5	36.2	C18 H19 N O6 Na

Elemental Composition Report



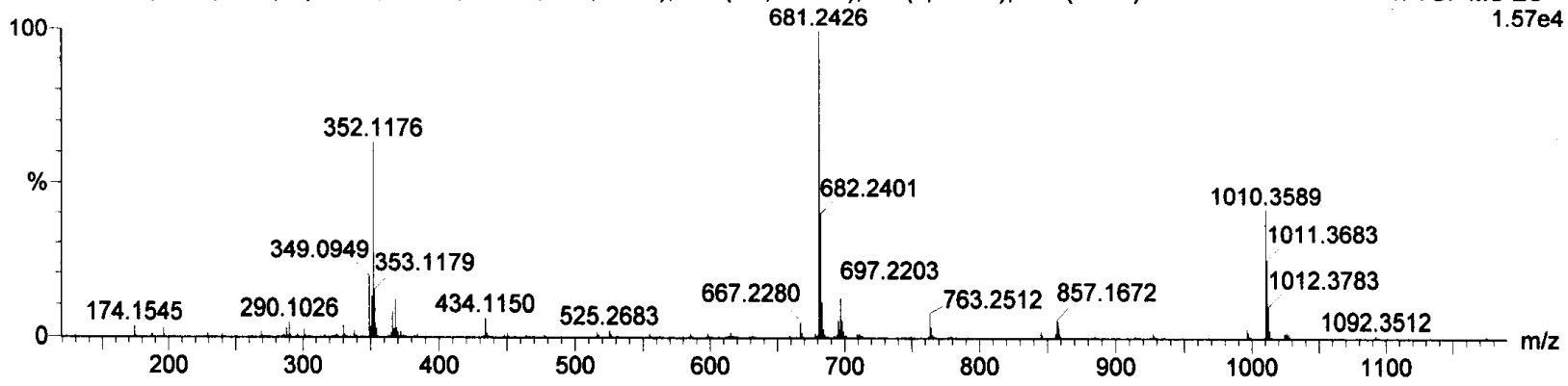
Single Mass Analysis (displaying only valid results)

Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100 H: 0-150 N: 1-1 O: 5-5 Na: 1-1

4alm
12092700 65 (1.658) AM (Top,4, Ar,5000.0,475.27,1.00,LS 10); Sm (Mn, 2x1.00); Sb (1,40.00); Cm (64:77)

09:17:43
1: TOF MS ES+
1.57e4

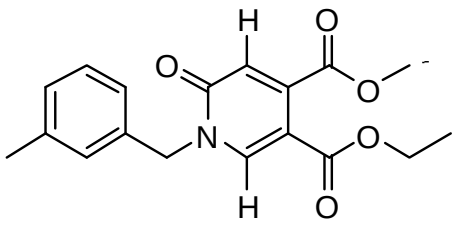


Minimum:				-200.0		
Maximum:		5.0	5.0	200.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
352.1176	352.1161	1.5	4.3	9.5	28.7	C18 H19 N O5 Na

Elemental Composition Report

Single Mass Analysis (displaying only valid results)

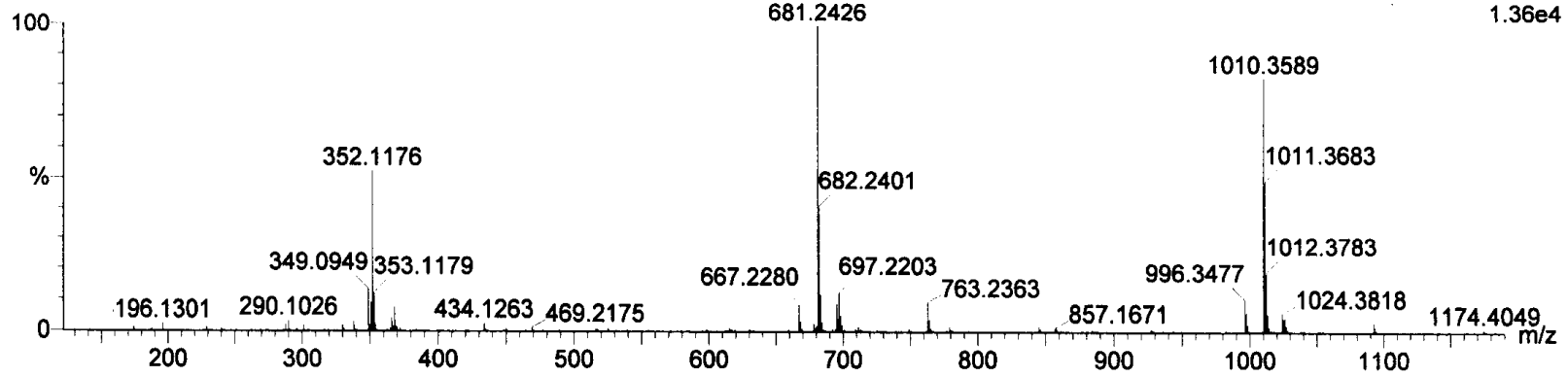
Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None



Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100 H: 0-150 N: 1-1 O: 5-5 Na: 1-1

4a\
12092701 34 (0.863) AM (Top,4, Ar,5000.0,475.27,1.00,LS 10); Sm (Mn, 2x1.00); Sb (1,40.00); Cm (15:34)

09:24:38
1: TOF MS ES+
1.36e4

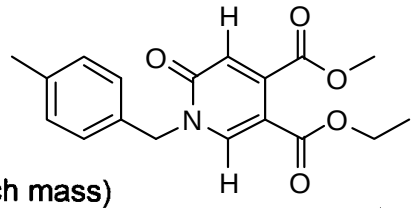


Minimum:				-200.0		
Maximum:		5.0	5.0	200.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
352.1176	352.1161	1.5	4.3	9.5	16.6	C18 H19 N O5 Na

Elemental Composition Report

Single Mass Analysis (displaying only valid results)

Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

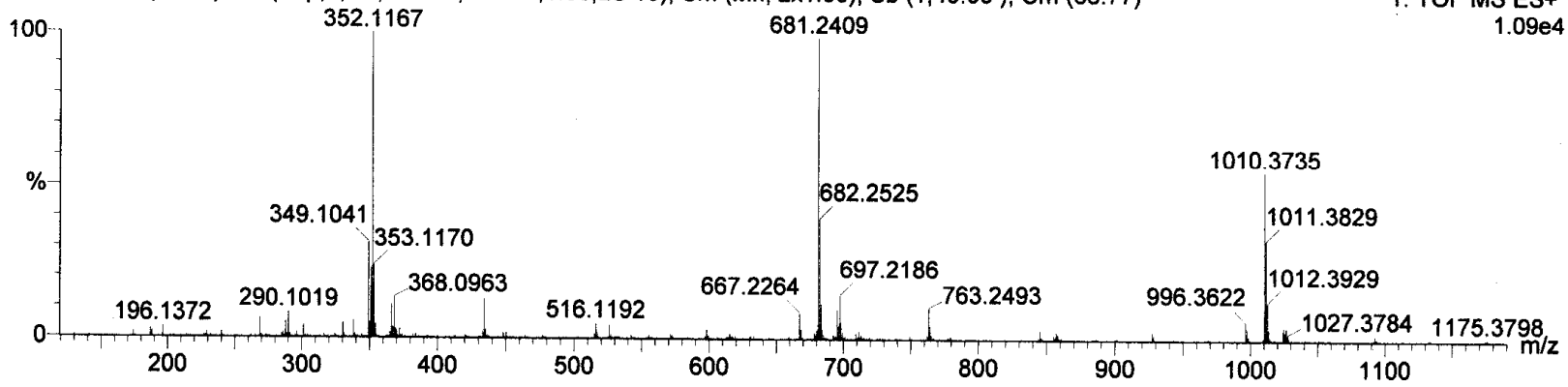


Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100 H: 0-150 N: 1-1 O: 5-5 Na: 1-1

4a

12092702 72 (1.829) AM (Top,4, Ar,5000.0,475.27,1.00,LS 10); Sm (Mn, 2x1.00); Sb (1,40.00); Cm (65:77)

09:31:14
1: TOF MS ES+
1.09e4

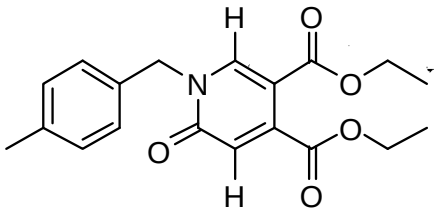


Minimum:				-200.0		
Maximum:		5.0	5.0	200.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
352.1167	352.1161	0.6	1.7	9.5	35.7	C18 H19 N O5 Na

Elemental Composition Report

Single Mass Analysis (displaying only valid results)

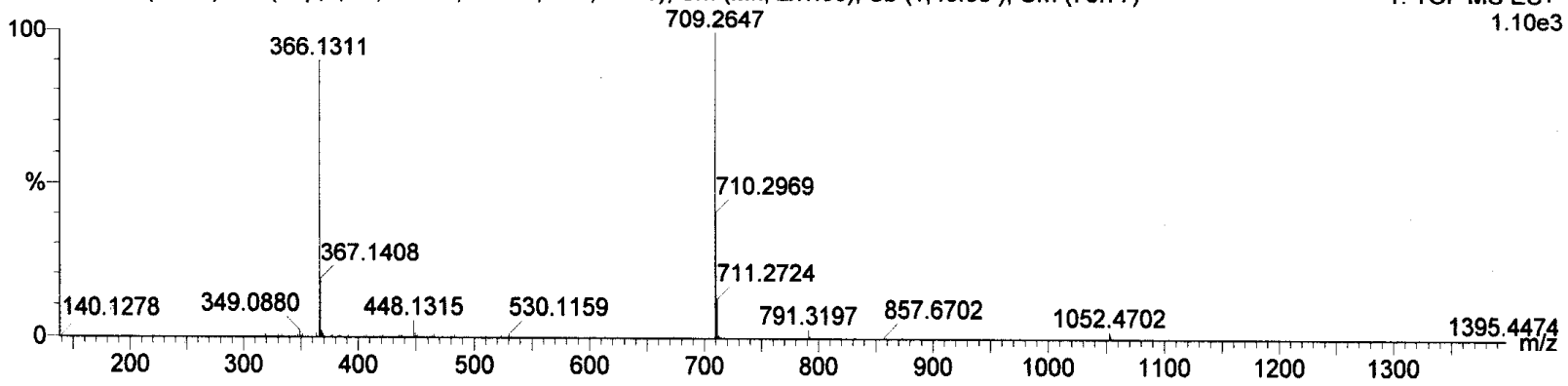
Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None



Monoisotopic Mass, Even Electron Ions
12 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100 H: 0-150 N: 1-1 O: 5-5 Na: 1-1

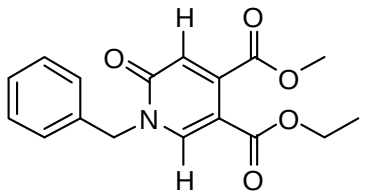
4a
12092647 77 (1.963) AM (Top,4, Ar,5000.0,475.27,1.00,LS 10); Sm (Mn, 2x1.00); Sb (1,40.00); Cm (76:77)

18:01:47
1: TOF MS ES+
1.10e3



Minimum:				-200.0		
Maximum:		5.0	5.0	200.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
366.1311	366.1317	-0.6	-1.6	9.5	6.7	C19 H21 N O5 Na

Elemental Composition Report



Single Mass Analysis (displaying only valid results)

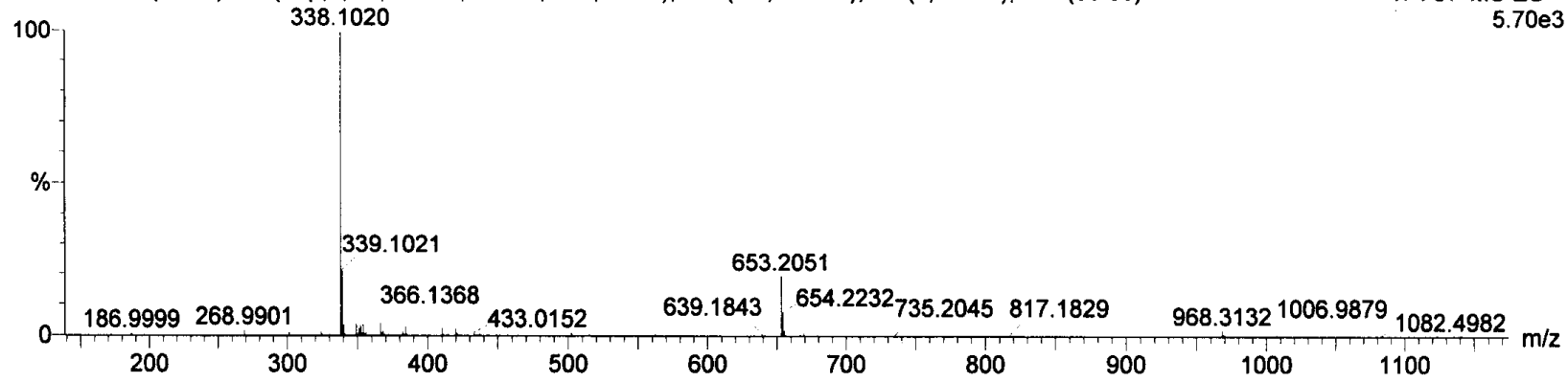
Tolerance = 50.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100 H: 0-150 N: 1-1 O: 5-5 Na: 1-1

4aq

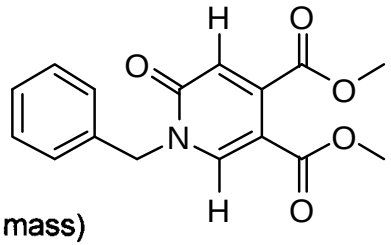
12092704 37 (0.967) AM (Top,4, Ar,5000.0,475.27,1.00,LS 10); Sm (Mn, 2x1.00); Sb (1,40.00); Cm (35:38)

09:49:49
1: TOF MS ES+
5.70e3



Minimum:				-200.0		
Maximum:		5.0	50.0	200.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
338.1020	338.1004	1.6	4.7	9.5	4.6	C17 H17 N O5 Na

Elemental Composition Report



Single Mass Analysis (displaying only valid results)

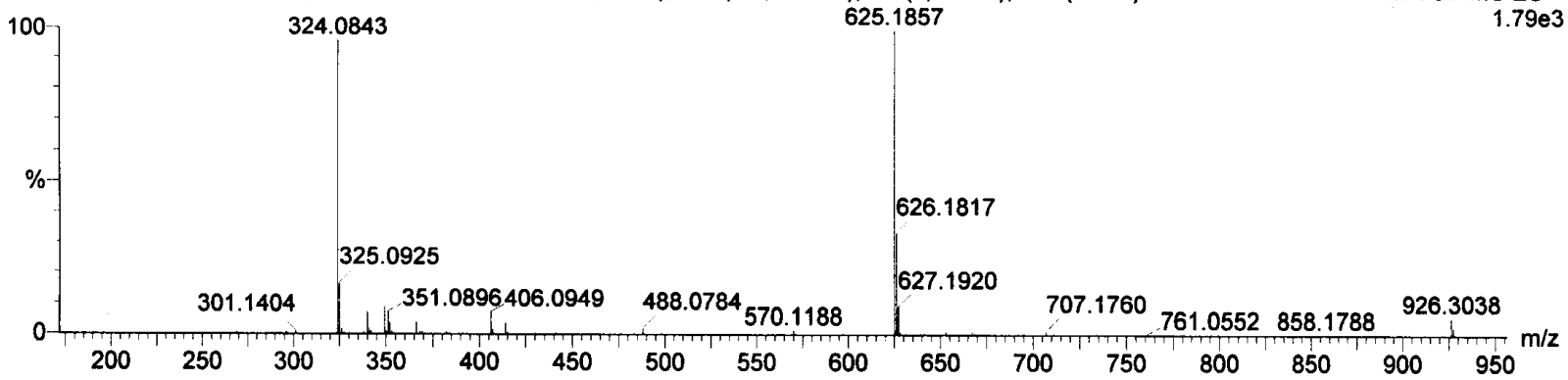
Tolerance = 50.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100 H: 0-150 N: 1-1 O: 5-5 Na: 1-1

4aq 4aY

12092705 42 (1.058) AM (Top,4, Ar,5000.0,475.27,1.00,LS 10); Sm (Mn, 2x1.00); Sb (1,40.00); Cm (41:42)

10:00:02
1: TOF MS ES+
1.79e3

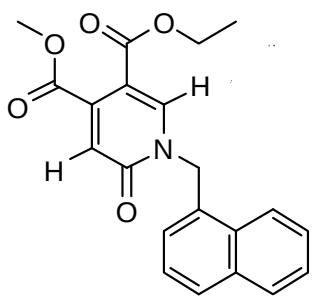


Minimum:				-200.0		
Maximum:		5.0	50.0	200.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
324.0843	324.0848	-0.5	-1.5	9.5	6.6	C16 H15 N O5 Na

Elemental Composition Report

Single Mass Analysis (displaying only valid results)

Tolerance = 50.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

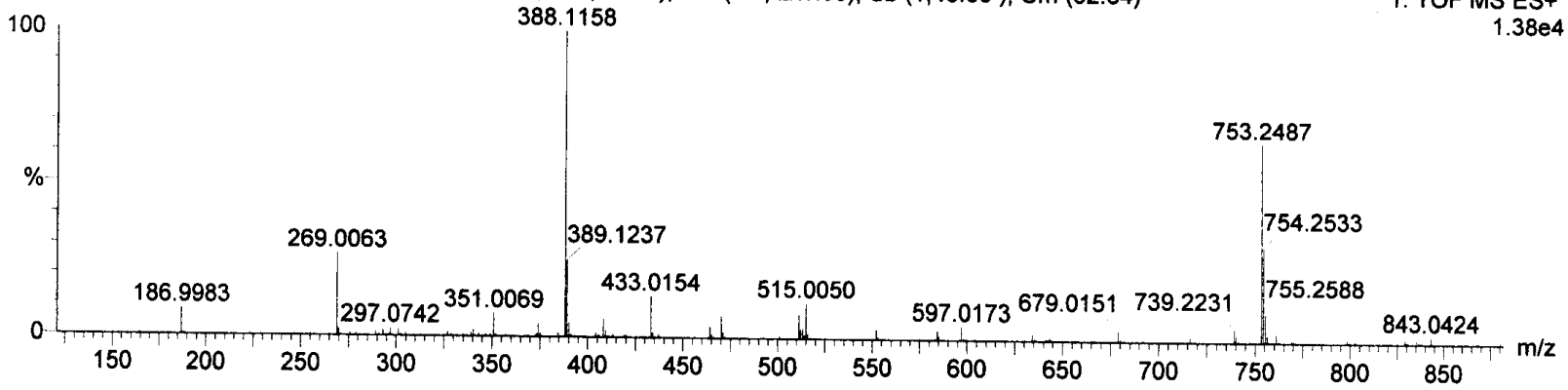


Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100 H: 0-150 N: 1-1 O: 5-5 Na: 1-1

4as

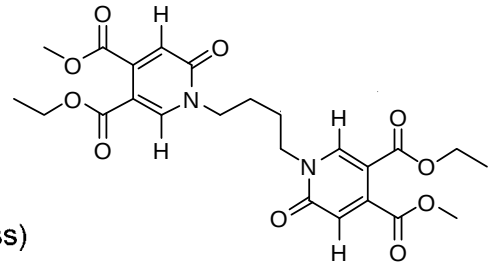
12120304 58 (1.485) AM (Top,4, Ar,5000.0,475.27,1.00,LS 10); Sm (Mn, 2x1.00); Sb (1,40.00); Cm (52:64)

10:08:48
1: TOF MS ES+
1.38e4



Minimum:				-200.0		
Maximum:		5.0	50.0	200.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
388.1158	388.1161	-0.3	-0.8	12.5	8.4	C21 H19 N O5 Na

Elemental Composition Report



Single Mass Analysis (displaying only valid results)

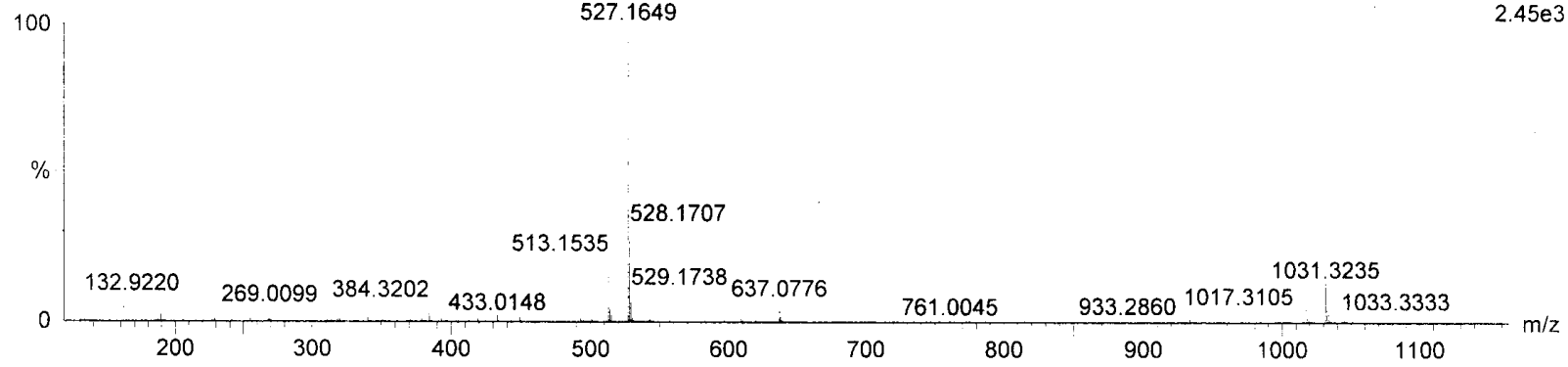
Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-120 H: 0-150 N: 2-2 O: 10-10 Na: 1-1

~~COX-11~~ 4at

13032812 21 (0.390) AM (Cen,2, 80.00, Ht,5000.0,0.00,1.00); Sm (Mn, 2x1.00); Cm (19:25)

11:22:07
1: TOF MS ES+
2.45e3

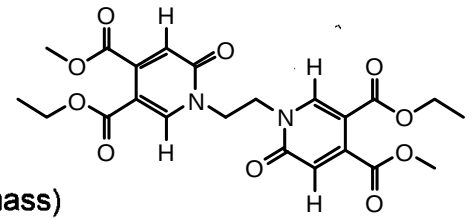


Minimum:				-200.0		
Maximum:		5.0	5.0	200.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
527.1649	527.1642	0.7	1.3	11.5	0.6	C24 H28 N2 O10 Na

Elemental Composition Report

Single Mass Analysis (displaying only valid results)

Tolerance = 50.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

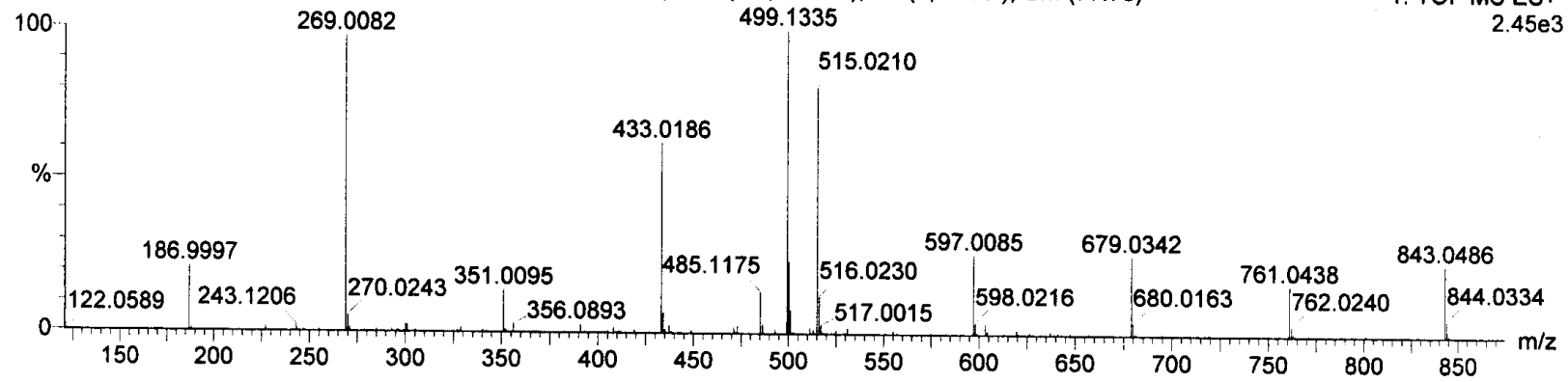


Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100 H: 0-150 N: 2-2 O: 10-10 Na: 1-1

4au

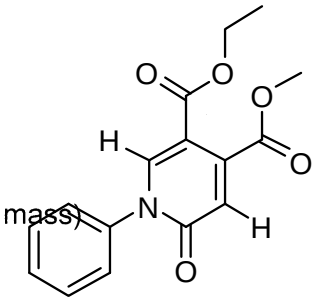
12120307 78 (1.970) AM (Top,4, Ar,5000.0,475.27,1.00,LS 10); Sm (Mn, 2x1.00); Sb (1,40.00); Cm (77:78)

10:30:22
1: TOF MS ES+
2.45e3



Minimum:				-200.0		
Maximum:		5.0	50.0	200.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
499.1335	499.1329	0.6	1.2	11.5	14.7	C22 H24 N2 O10 Na

Elemental Composition Report

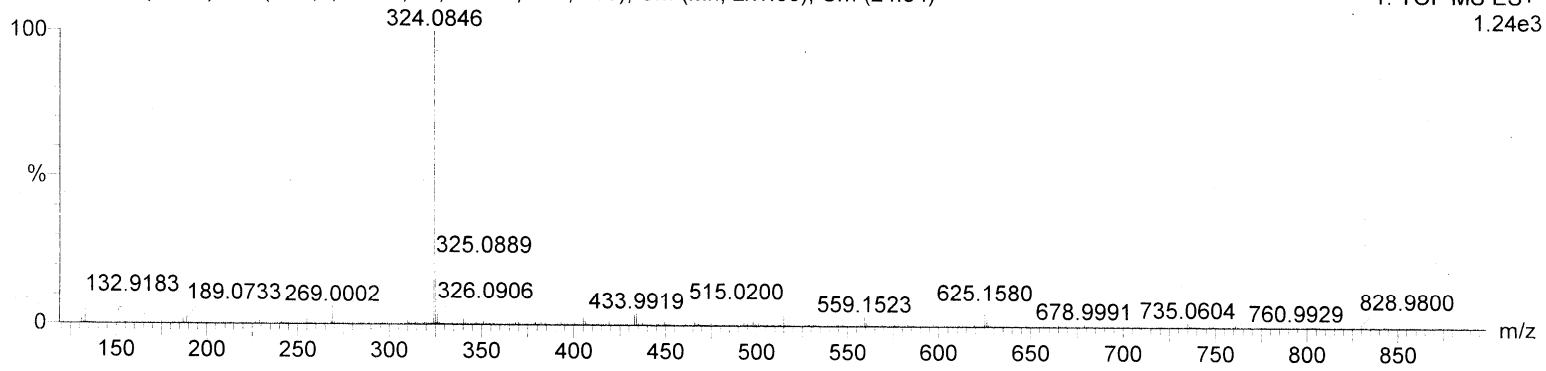


Single Mass Analysis (displaying only valid results)
Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-120 H: 0-150 N: 1-1 O: 5-5 Na: 1-1

~~COX-13~~ 4aV
13032814 29 (0.543) AM (Cen,2, 80.00, Ht,5000.0,0.00,1.00); Sm (Mn, 2x1.00); Cm (24:34)

12:04:26
1: TOF MS ES+
1.24e3

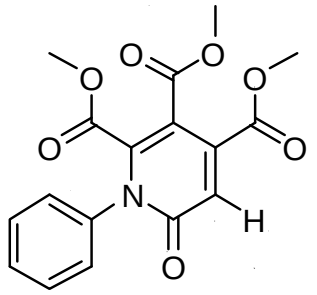


Minimum:				-200.0		
Maximum:		5.0	5.0	200.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
324.0846	324.0848	-0.2	-0.6	9.5	0.1	C16 H15 N O5 Na

Elemental Composition Report **Page 1**

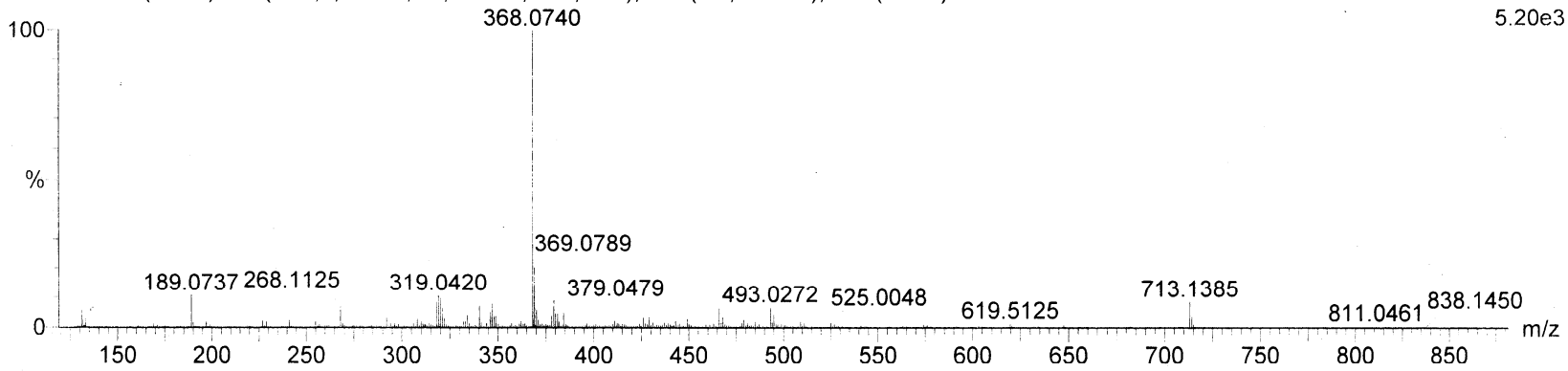
Single Mass Analysis (displaying only valid results)

Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None



Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-120 H: 0-150 N: 1-1 O: 7-7 Na: 1-1

CQX-1 09:59:45
13032802 51 (0.952) AM (Cen,2, 80.00, Ht,5000.0,0.00,1.00); Sm (Mn, 2x1.00); Cm (51:70) 1: TOF MS ES+
5.20e3

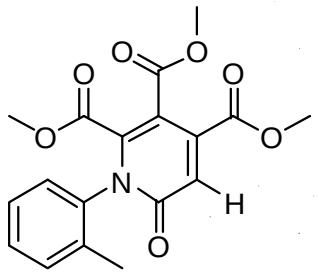


Minimum:				-200.0		
Maximum:		5.0	5.0	200.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
368.0740	368.0746	-0.6	-1.6	10.5	28.2	C17 H15 N O7 Na

Elemental Composition Report

Single Mass Analysis (displaying only valid results)

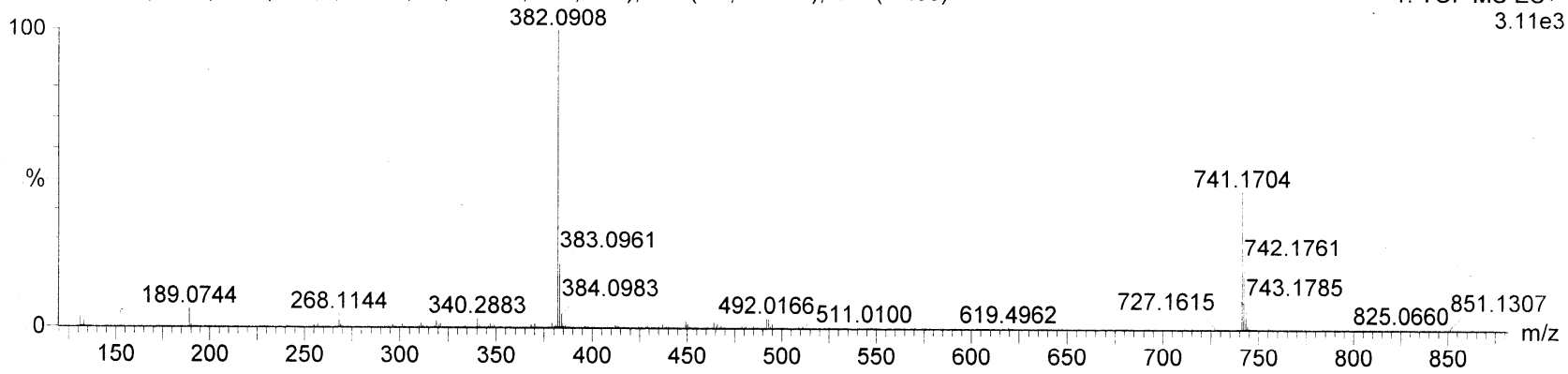
Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None



Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-120 H: 0-150 N: 1-1 O: 7-7 Na: 1-1

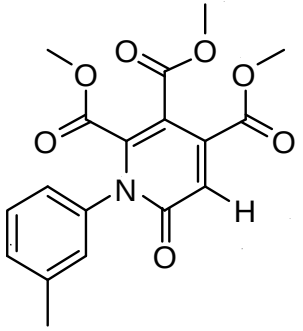
CQX-2
13032803 55 (1.023) AM (Cen,2, 80.00, Ht,5000.0,0.00,1.00); Sm (Mn, 2x1.00); Cm (44:55)

10:29:17
1: TOF MS ES+
3.11e3



Minimum:				-200.0		
Maximum:		5.0	5.0	200.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
382.0908	382.0903	0.5	1.3	10.5	1.9	C18 H17 N O7 Na

Elemental Composition Report

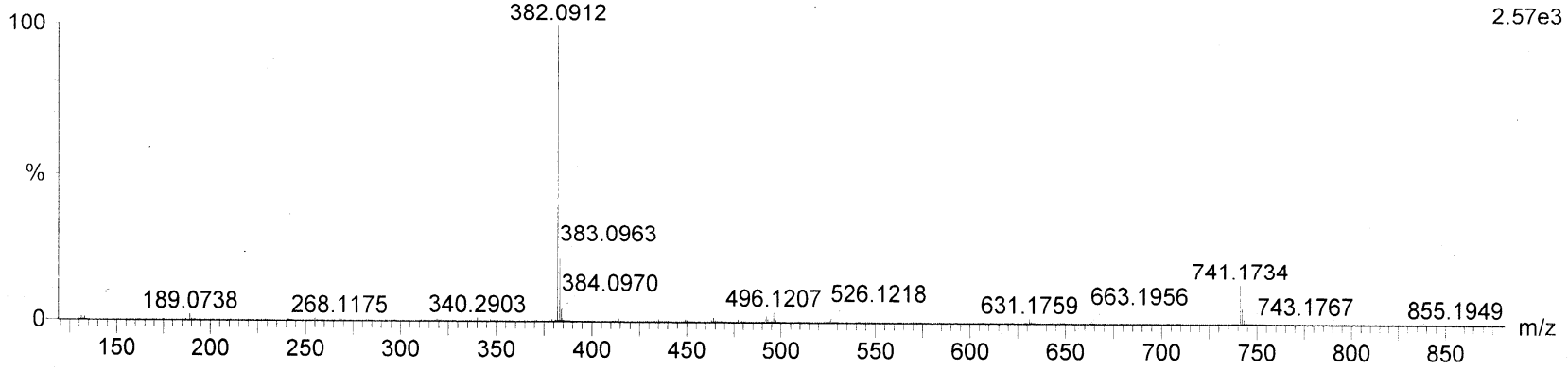


Single Mass Analysis (displaying only valid results)
Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-120 H: 0-150 N: 1-1 O: 7-7 Na: 1-1

CQX-3
13032804 57 (1.059) AM (Cen,2, 80.00, Ht,5000.0,0.00,1.00); Sm (Mn, 2x1.00); Cm (50:58)

10:35:17
1: TOF MS ES+
2.57e3

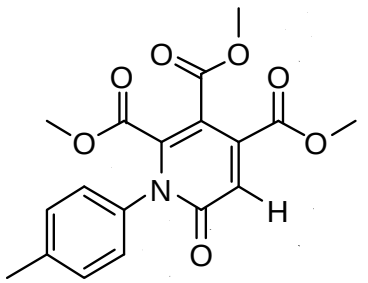


Minimum:				-200.0		
Maximum:		5.0	5.0	200.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
382.0912	382.0903	0.9	2.4	10.5	0.2	C18 H17 N O7 Na

Elemental Composition Report

Single Mass Analysis (displaying only valid results)

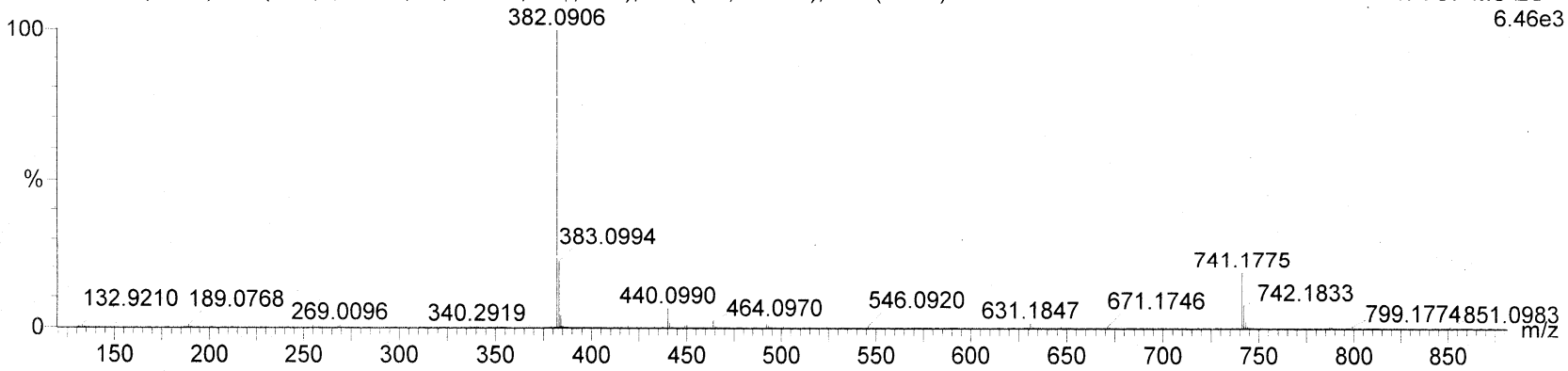
Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None



Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-120 H: 0-150 N: 1-1 O: 7-7 Na: 1-1

CQX-4
13032805 36 (0.669) AM (Cen,2, 80.00, Ht,5000.0,0.00,1.00); Sm (Mn, 2x1.00); Cm (28:36)

10:39:41
1: TOF MS ES+
6.46e3



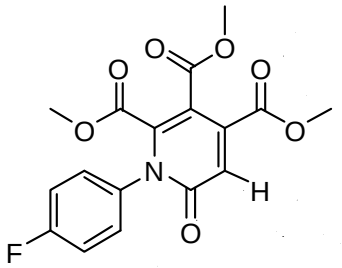
Minimum:				-200.0		
Maximum:		5.0	5.0	200.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
382.0906	382.0903	0.3	0.8	10.5	4.5	C18 H17 N O7 Na

Elemental Composition Report **Page 1**

Single Mass Analysis (displaying only valid results)

Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0

Selected filters: None



Monoisotopic Mass, Even Electron Ions

13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 0-120 H: 0-150 N: 1-1 O: 7-7 F: 1-1 Na: 1-1

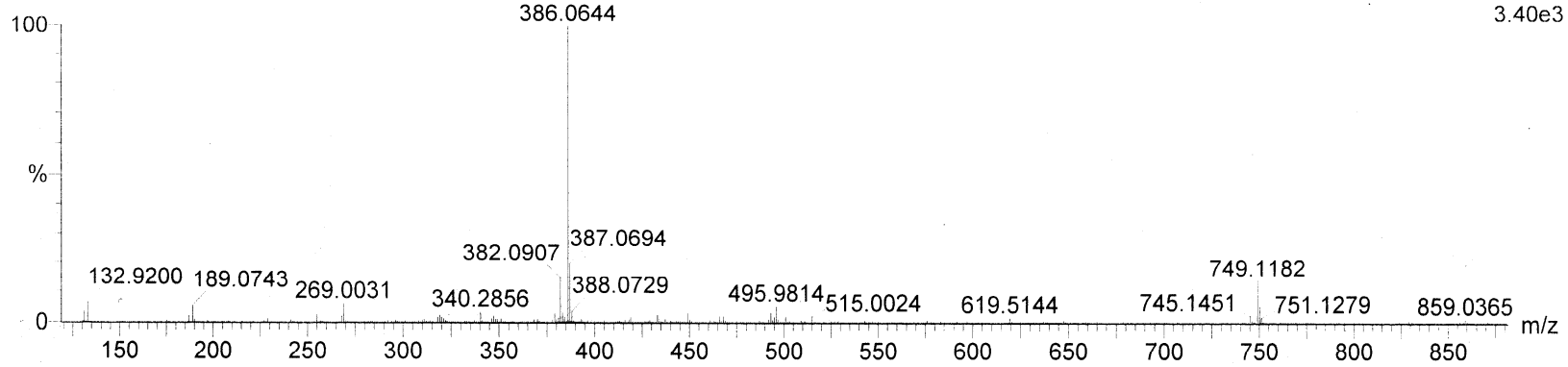
CQX-5

13032806 67 (1.249) AM (Cen,2, 80.00, Ht,5000.0,0.00,1.00); Sm (Mn, 2x1.00); Cm (53:70)

10:46:10

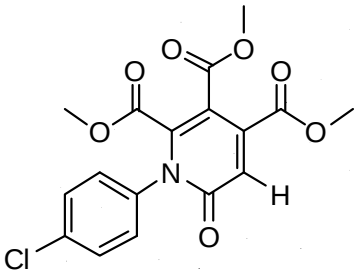
1: TOF MS ES+

3.40e3



Minimum:				-200.0		
Maximum:		5.0	5.0	200.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
386.0644	386.0652	-0.8	-2.1	10.5	0.2	C17 H14 N O7 F Na

Elemental Composition Report



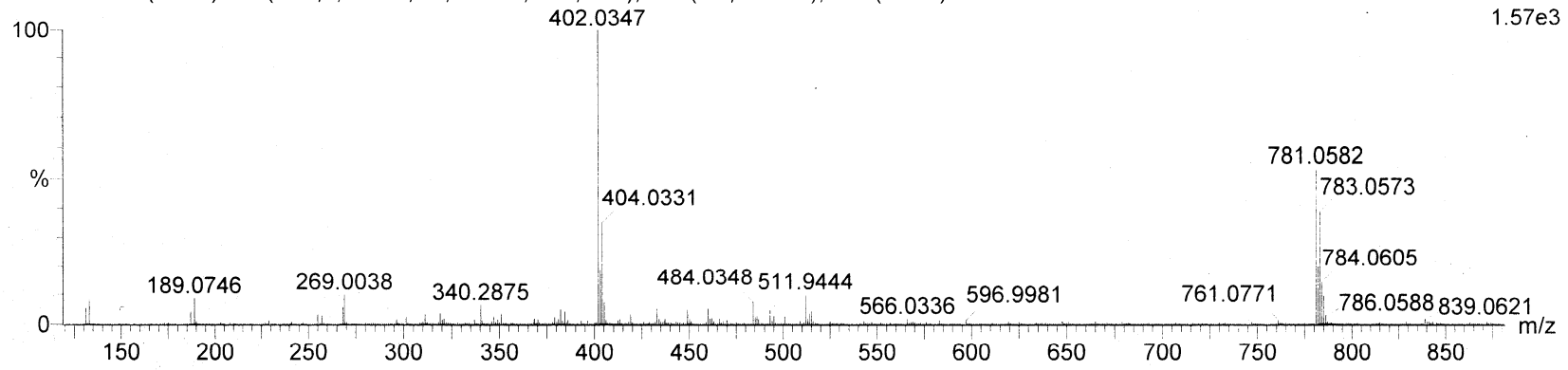
Single Mass Analysis (displaying only valid results)

Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-120 H: 0-150 N: 1-1 O: 7-7 Na: 1-1 Cl: 1-1

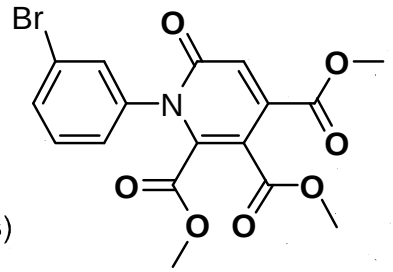
CQX-6
13032807 48 (0.891) AM (Cen,2, 80.00, Ht,5000.0,0.00,1.00); Sm (Mn, 2x1.00); Cm (41:52)

10:52:22
1: TOF MS ES+
1.57e3



Minimum:				-200.0		
Maximum:		5.0	5.0	200.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
402.0347	402.0356	-0.9	-2.2	10.5	0.4	C17 H14 N O7 Na Cl

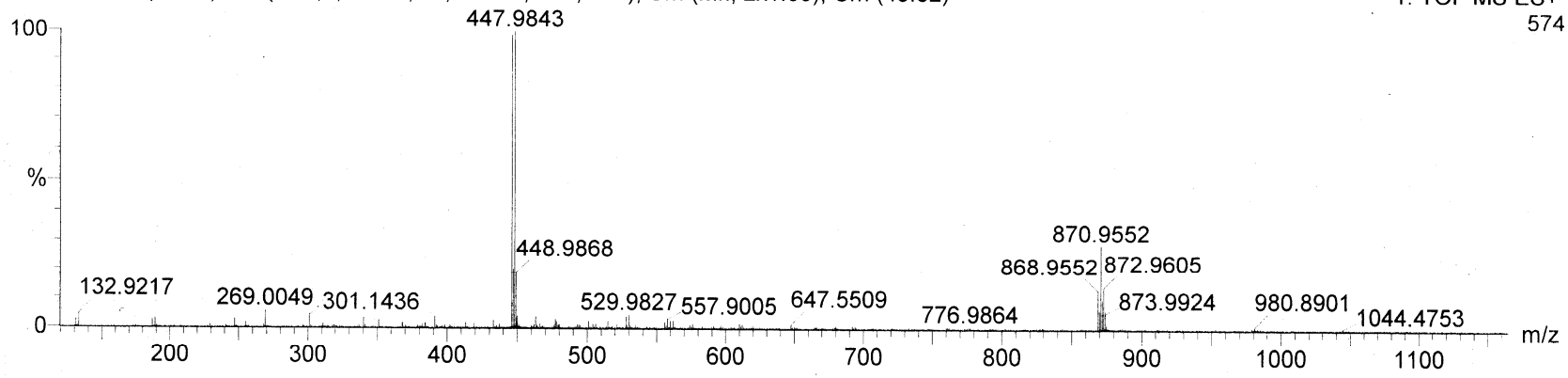
Elemental Composition Report



Single Mass Analysis (displaying only valid results)
Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-120 H: 0-150 N: 1-1 O: 7-7 Na: 1-1 Br: 1-1

CQX-7
13032808 51 (0.950) AM (Cen,2, 80.00, Ht,5000.0,0.00,1.00); Sm (Mn, 2x1.00); Cm (43:52)
10:57:16
1: TOF MS ES+
574

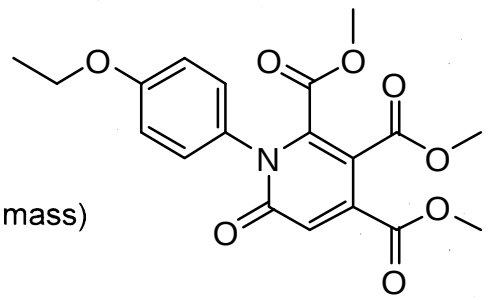


Minimum:				-200.0		
Maximum:		5.0	5.0	200.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
445.9858	445.9851	0.7	1.6	10.5	0.1	C17 H14 N O7 Na Br

Elemental Composition Report

Single Mass Analysis (displaying only valid results)

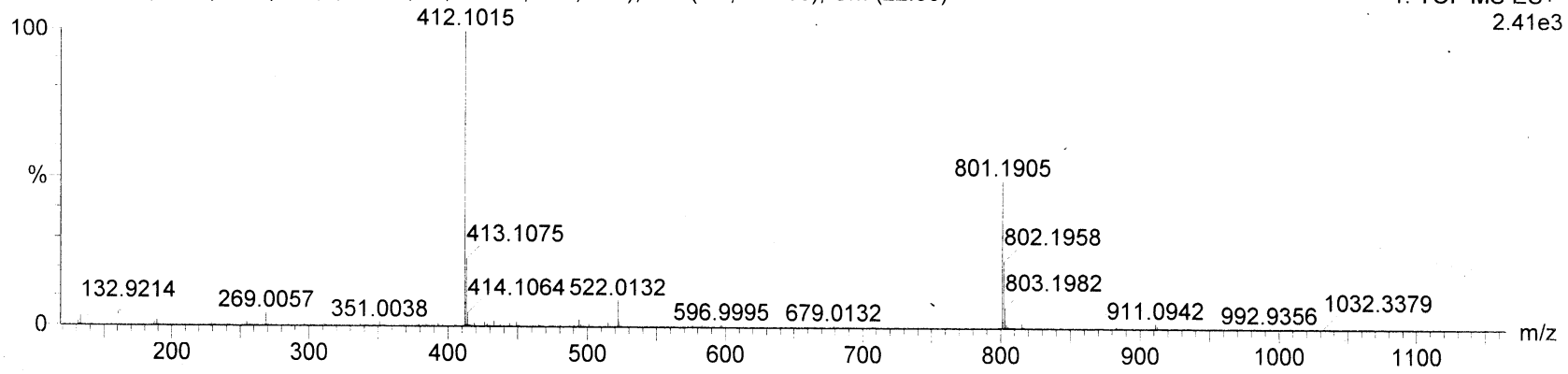
Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None



Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-120 H: 0-150 N: 1-1 O: 8-8 Na: 1-1

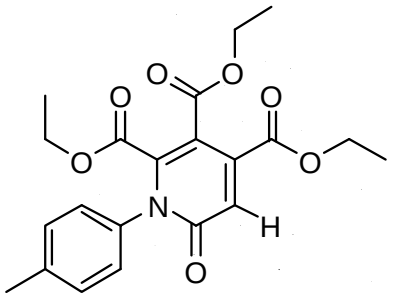
CQX-8
13032809 32 (0.594) AM (Cen,2, 80.00, Ht,5000.0,0.00,1.00); Sm (Mn, 2x1.00); Cm (22:33)

11:02:25
1: TOF MS ES+
2.41e3



Minimum:				-200.0		
Maximum:		5.0	5.0	200.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
412.1015	412.1008	0.7	1.7	10.5	0.6	C19 H19 N O8 Na

Elemental Composition Report



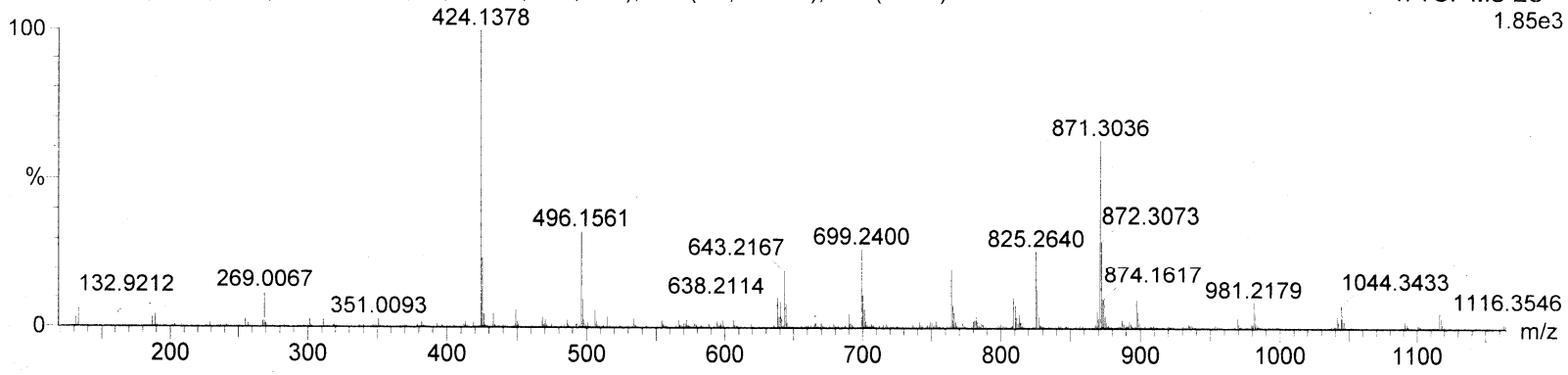
Single Mass Analysis (displaying only valid results)

Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-120 H: 0-150 N: 1-1 O: 7-7 Na: 1-1

CQX-9
13032810 59 (1.098) AM (Cen,2, 80.00, Ht,5000.0,0.00,1.00); Sm (Mn, 2x1.00); Cm (57:69)

11:09:22
1: TOF MS ES+
1.85e3

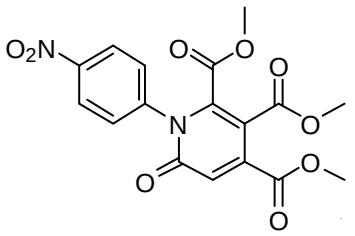


Minimum:				-200.0		
Maximum:		5.0	5.0	200.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
424.1378	424.1372	0.6	1.4	10.5	0.1	C21 H23 N O7 Na

Elemental Composition Report

Single Mass Analysis

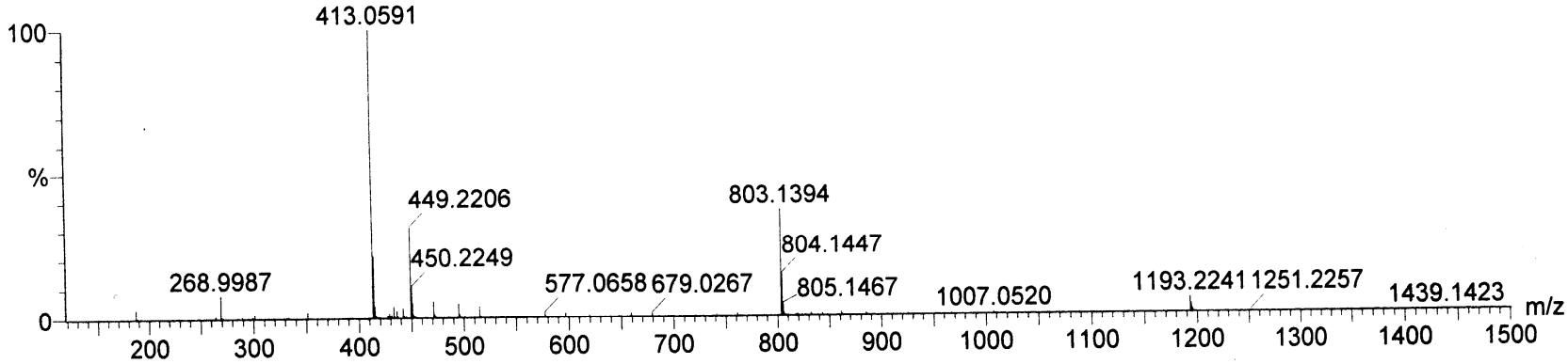
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 150.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3



Monoisotopic Mass, Even Electron Ions
4 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 0-150 H: 0-150 N: 2-2 O: 9-9 Na: 1-1

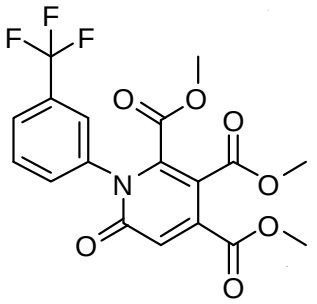
CQX-19
13042008 37 (0.614) AM (Cen,2, 80.00, Ht,5000.0,0.00,1.00); Sm (Mn, 2x3.00); Cm (27:39)

09:51:52
1: TOF MS ES+
2.97e+003



Minimum:				-1.5		
Maximum:		5.0	5.0	150.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
413.0591	413.0597	-0.6	-1.5	11.5	1.5	C17 H14 N2 O9 Na

Elemental Composition Report



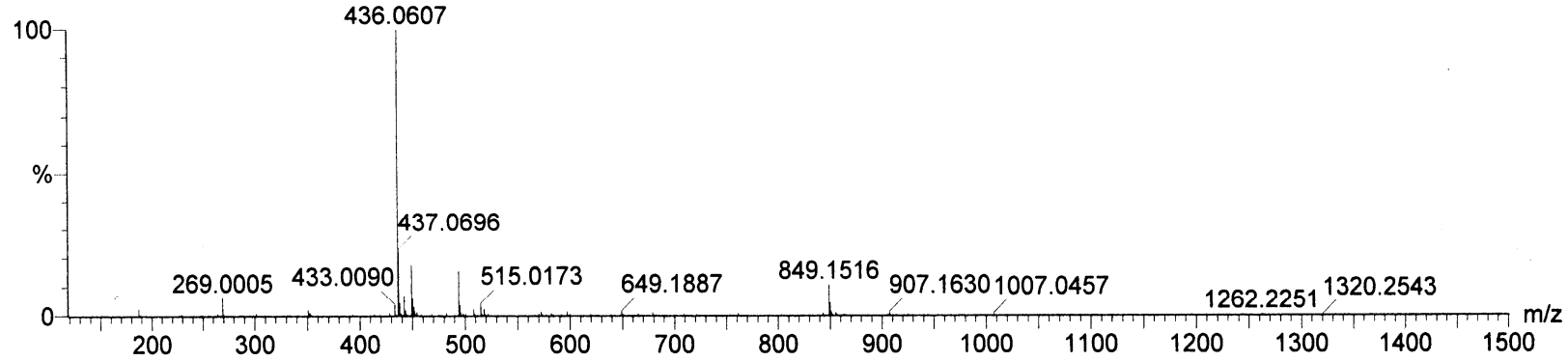
Single Mass Analysis

Tolerance = 50.0 PPM / DBE: min = -1.5, max = 150.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
4 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 0-150 H: 0-150 N: 1-1 O: 7-7 F: 3-3 Na: 1-1

CQX-1
13042007 2 (0.033) AM (Cen,2, 80.00, Ht,5000.0,0.00,1.00); Sm (Mn, 2x3.00); Cm (1:4)

09:43:19
1: TOF MS ES+
2.64e+003

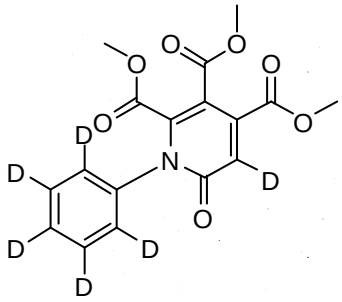


Minimum:				-1.5		
Maximum:		5.0	50.0	150.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
436.0607	436.0620	-1.3	-3.0	10.5	5.3	C18 H14 N O7 F3 Na

Elemental Composition Report

Single Mass Analysis (displaying only valid results)

Tolerance = 5.0 PPM / DBE: min = -200.0, max = 200.0
Selected filters: None

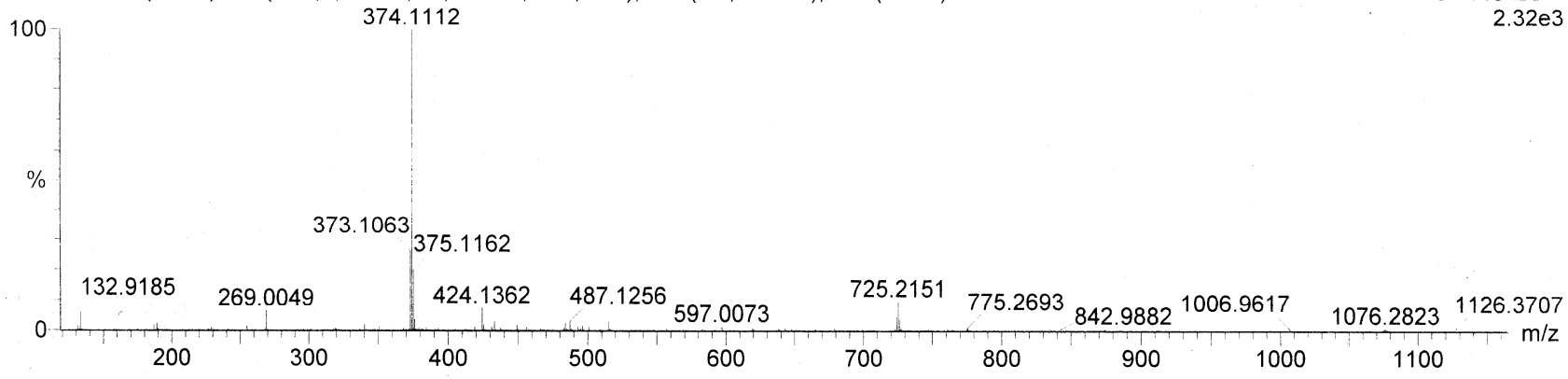


Monoisotopic Mass, Even Electron Ions
20 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-120 1H: 0-120 2H: 6-6 N: 1-1 O: 7-7 Na: 1-1

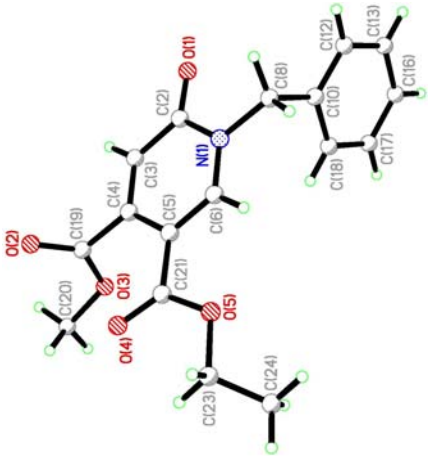
CQX-1

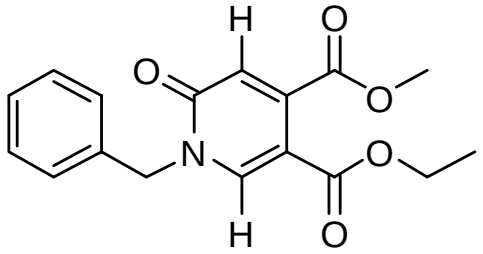
13032811 45 (0.837) AM (Cen,2, 80.00, Ht,5000.0,0.00,1.00); Sm (Mn, 2x1.00); Cm (41:47)

11:16:36
1: TOF MS ES+
2.32e3



Minimum:				-200.0		
Maximum:		5.0	5.0	200.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
374.1112	374.1123	-1.1	-2.9	10.5	188757.8	C17 1H9 2H6 N O7 Na





4aq

(CCDC 935076)

We used the slow evaporation method to grow the crystals of **4aq**. We first prepared a saturated solution of compound **4aq**, employing petroleum ether/ethyl acetate (4:1, v/v) as the solvent. Then the solution was transferred to a clean bottle and covered, with a few small holes in the cover. After that the bottle was placed in a quiet out of the way place. A few days later, the crystals of **4aq** were obtained. CCDC (**4aq**) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.